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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 70 Number 1

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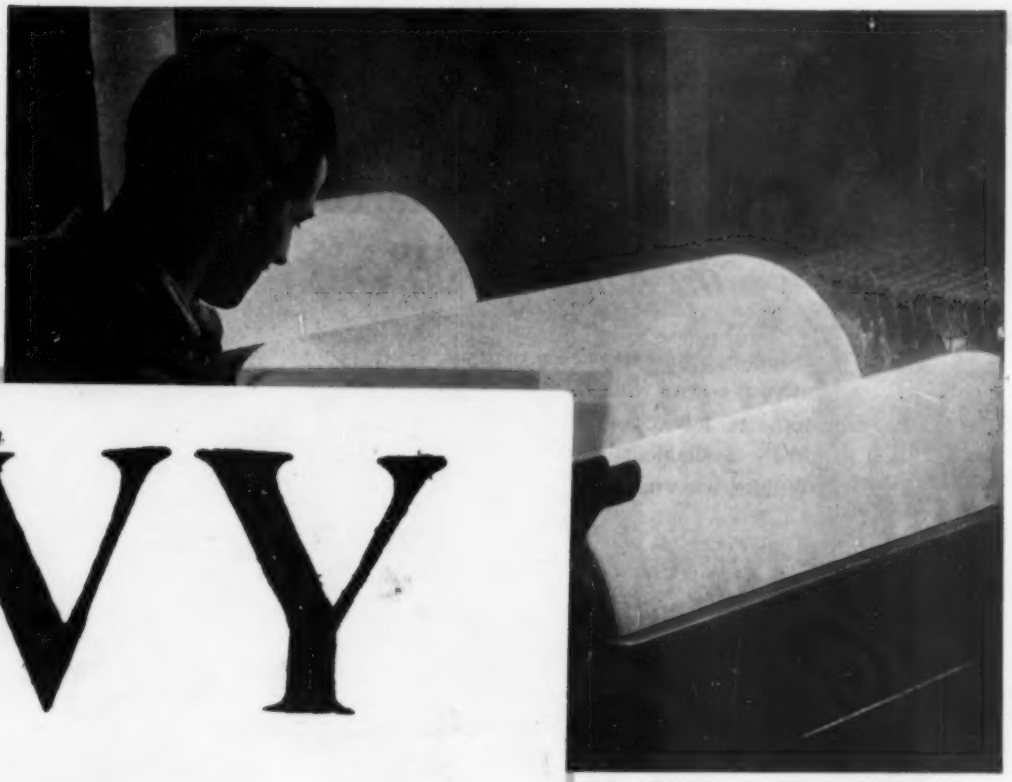

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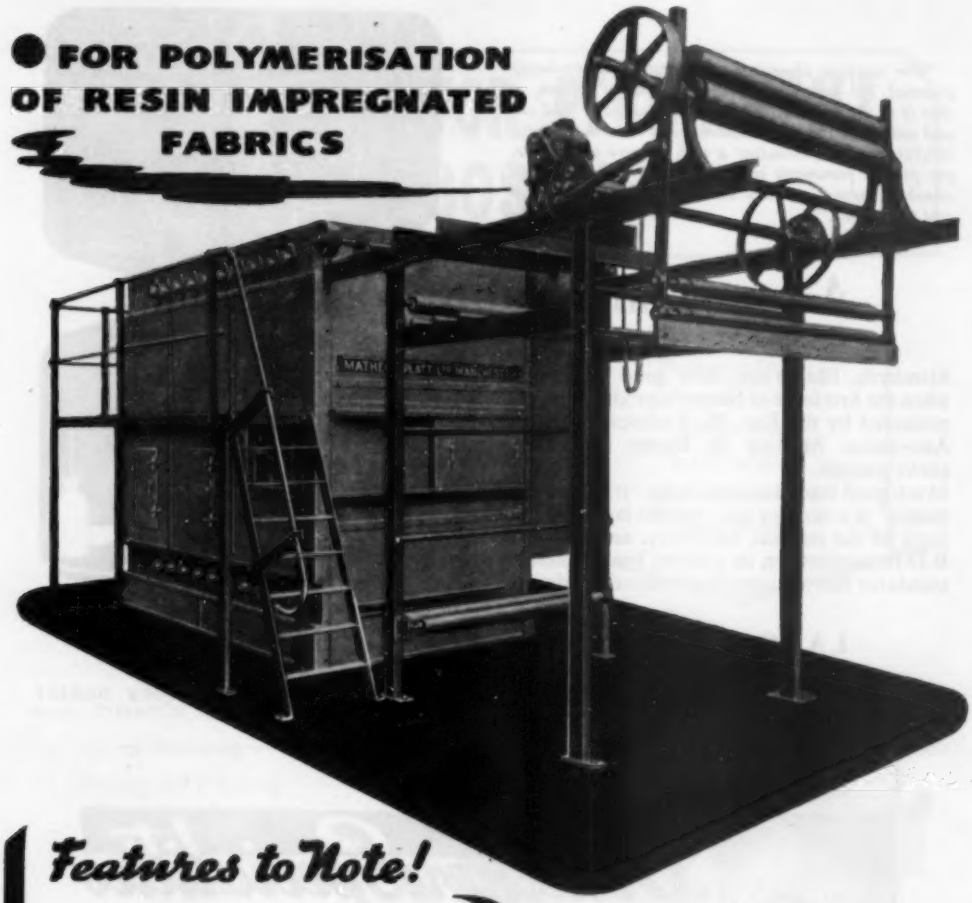

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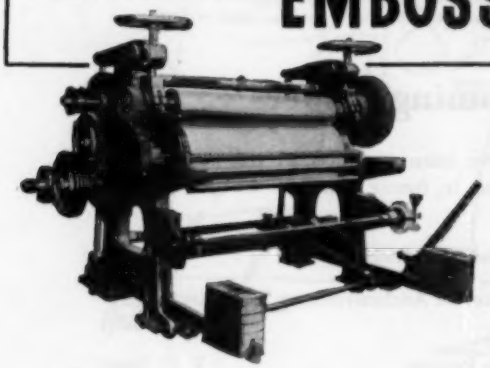

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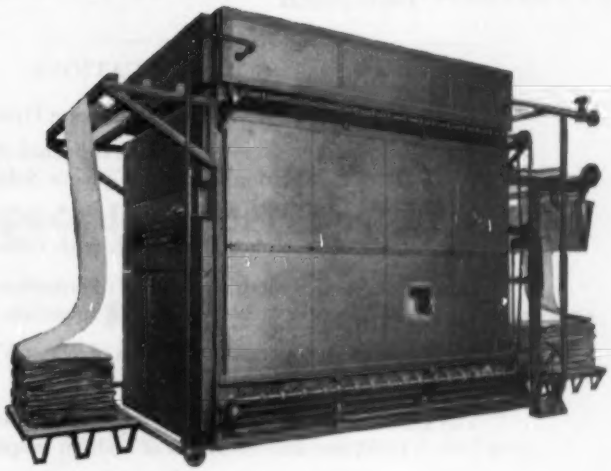

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1954 and pages 229–232 of the July 1953 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Experiments on the Chemistry of Anthraquinone Derivatives
W. Bradley

A Study of the Afterchrome Process *J. F. Gaunt*

Pressure Dyeing and Bleaching with special reference to Recent Developments *F. H. Marsh*

COMMUNICATIONS

The Dyeing of Acetate Rayon with Disperse Dyes.
I—Aqueous Solubility and the Influence of Dispersing Agents
II—The Relation between Aqueous Solubility and Dyeing Properties *C. L. Bird*

The Polarography of Azo Dyes *J. de O. Cabral and H. A. Turner*

Disulphide Bond Reactivity in Wool: Formation of Thiol Radicals on Exposure to Light *G. H. Crawshaw and J. B. Speakman*

The Effect of Dyeing on the Sorption of Water Vapour by Wool
P. Larose

The Effect on Wool of Boiling in Aqueous Solutions.
I—Water and Solutions of Sodium Sulphate at pH 1·5–9·0
R. V. Peryman

The Formation of Polymers in Wool
G. W. Madaras and J. B. Speakman

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FORTHCOMING MEETINGS OF THE SOCIETY

SCOTTISH SECTION

All Meetings in St. Enoch Hotel, Glasgow, at 7.0 p.m., unless otherwise stated

1954	
Tuesday 16th Feb.	LADIES' EVENING — Miss J. M. Young, B.A. (I.W.S.). *Fashion History in Wool.* Institution of Engineers and Shipbuilders, Scotland The lecture will be illustrated by a colour film strip, and a sound film *Paris, City of Fashion* will be shown also to supplement the lecture.
Tuesday 23rd March	ANNUAL GENERAL MEETING OF THE SECTION. To be followed by three short papers by Sectional members — details to be announced later

MIDLANDS SECTION

Except when stated otherwise, Lecture Meetings commence at 7.0 p.m.

1954	
Wednesday 24th Feb.	Douglas Haigh, Esq., A.T.I. *Some recent researches in the Dyeing of Felt.* Masonic Hall, Nuneaton
Thursday 4th March	R. F. Wilson, Esq. (B.C.C.) (Joint meeting with the Nottingham Textile Society). *Colour.* Gas Theatre, Nottingham
Wednesday 17th March	G. H. Lister, Esq., B.Sc., Ph.D. *The Migration Properties of Acid and Chrome Dyes.* Victoria Station Hotel, Nottingham
Friday 26th March	MIDLANDS SECTION DINNER. King's Head Hotel, Loughborough
Wednesday 7th April	ANNUAL GENERAL MEETING OF SECTION. J. A. Potter, Esq., M.A. *Dyeing and Finishing Synthetic Fibres in the Hosiery Industry.* King's Head Hotel, Loughborough
Friday and Saturday 28th - 29th May	Exhibition *Scientific Aids to the Dyeing Industry.* College of Technology, Leicester (*Details later*)

NORTHERN IRELAND SECTION

All Meetings to be held in Royal Avenue Hotel, Belfast, at 7.30 p.m.

1954	
Friday 5th Feb.	W. Ford-Kirkpatrick, Esq. *Putting the Colour into Photography*
Friday 5th March	J. Potter, Esq. (Clayton Aniline Co. Ltd.). *Special Finishes for Textiles and Their Evaluation.* (Joint meeting with Foremen Dyers Guild)
Late March or early April	ANNUAL GENERAL MEETING AND DINNER. (*Details later*)

MANCHESTER JUNIOR BRANCH

All lectures to be held in the Reynold's Hall, Manchester College of Technology, at 6.30 p.m.

1954	
Monday 1st Feb.	C. L. Bird, Esq. *Theoretical Aspects of the Dyeing of Cellulose Acetate Rayon*
Monday 8th March	D. G. Evans, Esq., B.Sc. *Cibalan Dyes — Nylons and Nylon Unions*

WEST RIDING SECTION

Meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1954	
Thursday 11th Feb.	F. H. Marsh, Esq., B.Sc., F.R.I.C. (Longclose Engineering Co. Ltd.). *Modern Trends in the Manufacture of Dyeing Machinery with Particular Reference to High Temperature Dyeing*
Thursday 25th Feb.	A. R. Smith, Esq., B.A., B.Sc., A.R.I.C. (British Industrial Plastics Ltd.). *The Application of Resins to Textiles*

WEST RIDING SECTION (*continued*)

Thursday 11th March	Prof. J. B. Speakman (The University, Leeds). Lecture. Title to be announced later
Thursday 25th March	LECTURE AND ANNUAL GENERAL MEETING
Tuesday 12th Jan.	S. Kershaw, Esq., F.T.I. (By invitation of the Yorkshire Section of the Textile Institute). *Experiences on a United Nations Assignment.* Midland Hotel, Bradford, at 7.15 p.m.
Monday 8th March	G. Kenneth Seddon, Esq. (By invitation of the Bradford Textile Society). *Interesting Points in Dyeing and Finishing.* Midland Hotel, Bradford, at 7.15 p.m.

LONDON SECTION

All Meetings will be held at the Royal Society, Burlington House, Piccadilly, London W.1, at 6.0 p.m. unless otherwise stated

1954	
Friday 5th Feb.	C. P. Tattersfield (Courtaulds Ltd.). *Dyeing and Finishing in relation to Use-Development Work*
Friday 5th March	*The London Lecture.* Subject, Speaker and venue to be given

HUDDERSFIELD SECTION

All Lectures at Field's Cafe, Westgate, Huddersfield, at 7.30 p.m. unless otherwise stated

1954	
Tuesday 16th Feb.	J. G. Grundy, Esq. (Clayton Aniline Co. Ltd.). *Cibalan Dyes and their Fastness Properties on Nylon and Application to Wool-Nylon Unions*
Tuesday 16th Mar.	ANNUAL GENERAL MEETING, followed by a lecture, *Fastness Properties for Users Satisfaction.* J. S. Ingham, Esq. (Marks & Spencer Ltd., London)

BRADFORD JUNIOR BRANCH

All Meetings held at Bradford Technical College commencing at 7.15 p.m. unless otherwise stated

1954	
Thursday 4th Feb.	C. L. Bird, Esq., M.Sc., F.R.I.C. *Theoretical Aspects of the Dyeing of Cellulose Acetate*
Tuesday 16th Feb.	Film Evening. Mather & Platt
Thursday Afternoon 18th Feb.	Visit to Messrs. J. Hoyle & Son Ltd., Huddersfield
Thursday 25th Feb.	Junior Branch Dance
Friday 12th March	Students' Competition Evening

LEEDS JUNIOR BRANCH

All meetings held in the Colour Chemistry Lecture Theatre, The University, Leeds 2, on Tuesdays at 3.30 p.m.

1954	
Tuesday 2nd Feb.	Dr. P. Standring (Messrs. I.C.I. Ltd.). *The Chemistry and Dyeing of "Terylene"* (To be confirmed)
Tuesday 16th Feb.	M. R. Fox, Esq. (Messrs. I.C.I. Ltd.). *Experiments on the Continuous Dyeing of Wool with Special Reference to the Standfast Molten Metal Technique*
Tuesday 9th Mar.	Dr. G. H. Lister (Messrs. Sandoz Products Ltd.). *Migration Properties of Acid and Chrome Dyes*

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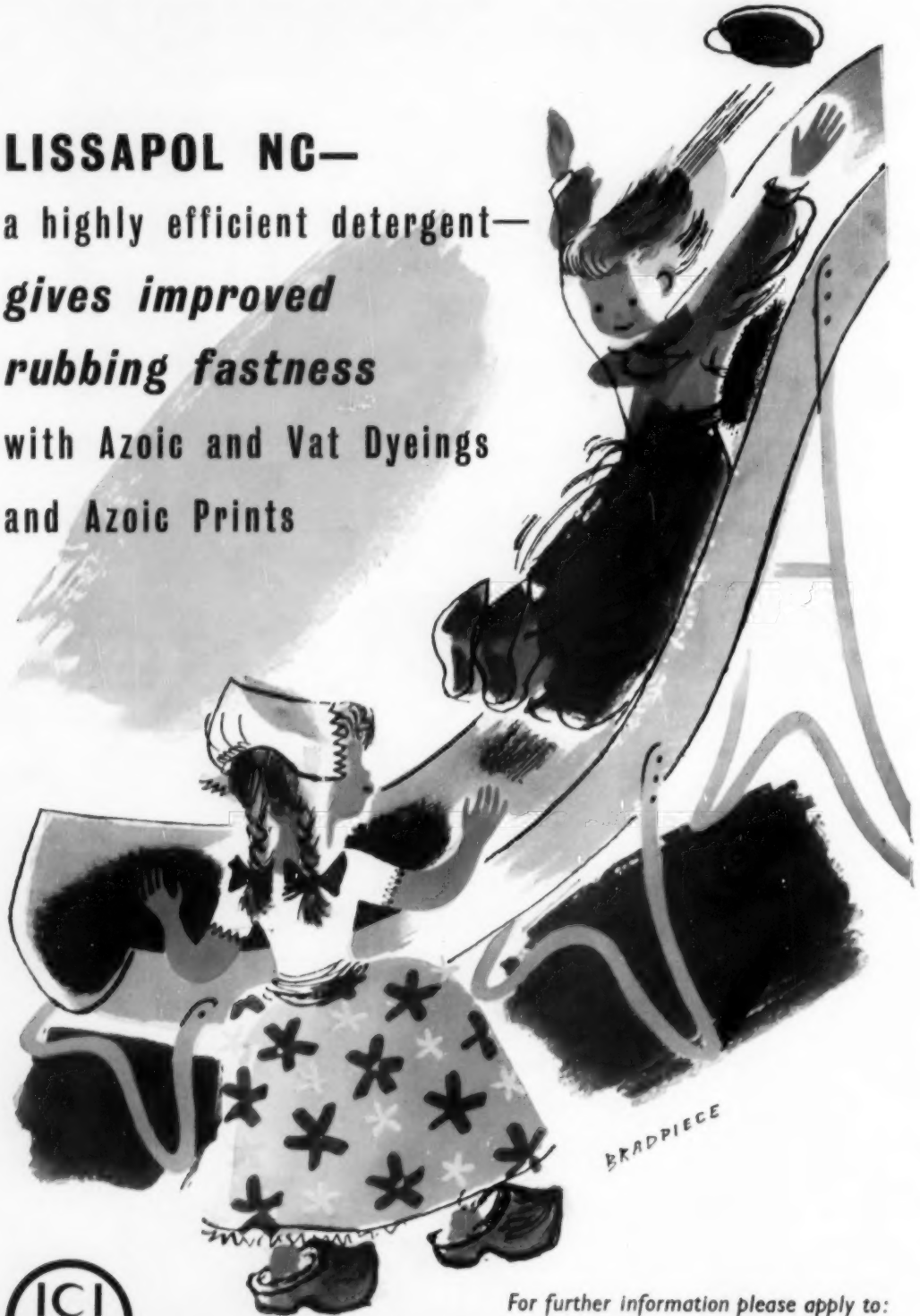

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THE JOURNAL
OF THE
Society of Dyers and Colourists

Volume 70 Number 1 JANUARY 1954 *Issued Monthly*

OFFICIAL NOTICES

OBJECTS OF THE SOCIETY OF DYERS AND COLOURISTS

To promote the advancement of science and technology, especially in the theory and practice of the tinctorial arts.

To provide means for the wider dissemination and interchange of knowledge concerning the science and technology of colour and colouring matters, of their methods of application, and of the materials to which they may be applied.

To encourage education and research in any or all of the above subjects.

And for the furtherance of these objects—

To hold meetings for the reading of papers, for lectures, and for discussions.

To publish a *Journal*.

And to do all such other things as may be conducive to the attainment of these objects.

ELECTION OF OFFICERS AND MEMBERS OF COUNCIL

Under the revised Bye-laws which are now operative, Officers and Members of Council—other than the President and President-elect, who are nominated by Council and elected at the Annual General Meeting—are to be elected by a Postal Ballot of the members.

The attention of members is drawn to Bye-laws No. 12–21 inclusive and 22–26 inclusive, which relate to Officers and Members of Council respectively.

Nominations to be valid must be received by the Honorary Secretary of the Society at least six weeks prior to the date of the Annual General Meeting, which is on 30th April 1954.

MEMBERS AND JUNIOR MEMBERS

Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the General Secretary or from the Honorary Secretary of any Section of the Society. Applications must be proposed and seconded by members to whom the applicants are known personally. The rates of annual subscription are as follows—

(i) During the period of "full-time national service" the annual subscription to be waived entirely. Such members will be asked whether they wish to receive the *Journal*.

(ii) Ordinary Members between the ages of 21 and 25 years who are students, i.e. who are pursuing an accepted course, full or part time, at a recognised technical college or university and vouched for by the head of their department or other responsible person—15s. 0d.

(iii) Other Ordinary Members between the ages of 21 and 25 years—£2 2s. 0d.

(iv) Members having had forty years' continuous membership of the Society to be given the option of paying half the current annual subscription (vi) for Ordinary Members.

(v) Members having had fifty years' continuous membership of the Society—the annual subscription to be waived entirely.

(vi) All other Ordinary Members—£3 3s. 0d.

(vii) All other Junior Members—15s. 0d.

The *JOURNAL*

The *Journal* is posted free to all Members. Non-members can obtain copies on application to the Offices of the Society. All orders must be accompanied by a remittance of 6s. 8d. per copy (£4 0s. 0d. per annum post free). Back numbers of most issues of the Society's *Journal* can be supplied.

Members are reminded that under Bye-law 41 the *Journal* will not be forwarded to those who have not paid their subscription by 30th June 1954.

Members residing abroad are particularly requested to inform the General Secretary by separate post when sending their subscriptions by Money Order.

Communications on any subject related to the objects of the Society, especially such as are of an original character, are invited for consideration for publication in the *Journal*. Such Communications in the first instance should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.

General communications, including inquiries or orders for advertisements, should be addressed to the Offices of the Society, to which address all remittances should be sent.

REPRINTS OF LECTURES AND COMMUNICATIONS

Reprints of all lectures and communications are available after publication to members of the Society and non-members. The charges (postage included) are as follows—Single copies 2s. 6d. each; per dozen copies up to and including 8 pages, 12s. 6d., and for papers occupying more than 8 pages of the *Journal*, 17s. Orders should be addressed to "The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1". They can be accepted only if accompanied by remittance and if received immediately after publication of the paper.

ABSTRACTS SECTION

Attention is drawn to the fact that copies of the Abstracts Section of the *Journal* printed on only one side of the paper, so that individual abstracts can be cut out and pasted on cards, are available at a charge of 30s. 0d. per annum. Orders should be sent to the Society's offices at 19 Piccadilly, Bradford 1, Yorkshire.

FREE ADVERTISEMENTS

For the convenience of members the Publications Committee allows a limited number of advertisements relating to SITUATIONS WANTED to be inserted in the *Journal gratis*. Such advertisements must not exceed 24 words in length.

Replies may be addressed, *Box—, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.*

NOTICE TO AUTHORS OF PAPERS

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essentia that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. Centre headings should be employed sparingly. Side headings should be indented and underlined, and run into the text to which they apply by means of a dash. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the Index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and they should be indicated by small circles rather than by crosses in the case of a single graph, but where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

Twenty-five free copies of a reprint are supplied to the author of an original paper published in the *Journal*, or fifty free copies are supplied when there are two or more authors, and a further number may be purchased from the Society at the rates given on p. 1.

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The Society subscribes to the Royal Society Fair Copying Declaration (J.S.D.C., **66**, 54 (Jan. 1950); **67**, 236 (June 1951)).

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Many of the books and periodicals reviewed or abstracted in the *Journal* since 1948 (and in some cases earlier) are retained by the Society, and may be borrowed by members. Enquiries and applications should be directed to the Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.

LIBRARY OF THE CHEMICAL SOCIETY—LOAN OF BOOKS

Members of the Society may borrow books from the very extensive collection of works on pure and applied chemistry in the Library of the Chemical Society, by applying direct to *The Librarian, Chemical Society, Burlington House, Piccadilly, London W.1 (REGent 0675–6), and referring to their membership of the Society of Dyers and Colourists*. Letters on the subject should *not* be addressed to the Offices of the Society in Bradford.

DEPOSIT OF SEALED COMMUNICATIONS

I—The Society is prepared to receive from members and others and to keep as deposits, Sealed Communications dealing with any subject relating to the theory or practice of the Dyeing, Printing, and kindred industries.

II—Every deposit must bear on the cover a title for classification, the author's name, the date, and must be secured by a distinctive seal.

III—The deposit should be written in English, and, if it deals with machinery, be accompanied by sketches or drawings, or, in the case of dyeing or printing processes, by patterns.

IV—Every deposit will, unopened, be signed and sealed by the Honorary Secretary of the Society, immediately after receipt, and countersigned by another member of the Council.

V—The deposits will be numbered and entered into a special register, and an official receipt will be sent to the author.

VI—No charge will be made for registration and deposit.

VII—The deposits will be kept by the Society for a period of seven years. At the end of this period they will be opened and the contents read before the next meeting of the Council of the Society, and the contents, or an abstract thereof, may be published in the *Journal* of the Society at the discretion of the Publications Committee.

VIII—The author shall have the right to recall his deposit unopened at any time within the said seven years, or he may, at any time, order it to be opened and read before one of the Society's meetings. Such instructions must be accompanied by the official receipt.

IX—The Society will take every reasonable care of the deposits, but cannot be held responsible for their loss.

MEDALS AND AWARDS

HONORARY MEMBERS OF THE SOCIETY

1886–1915	*Sir H. E. Roscoe, M.P.	1931–1933	*Alfred Rée, Ph.D.
1902–1926	*F. H. Bowman, D.Sc., F.I.C., F.R.S.E.	1932–1940	*W. M. Gardner
1908–1916	*Prof. Dr. C. Graebe	1934–1947	*George Douglas
1908–1915	*Prof. Dr. C. Liebermann	1934–1946	*H. Grandage
1911–1916	*Prof. Adolf von Baeyer	1934–1940	*Christopher Rawson
1914–1924	*Count Hilaire de Chardonnet	1941–1948	*Prof. E. C. C. Baly, C.B.E., M.Sc., F.R.S.
1917–1941	*Arthur G. Green, M.Sc., F.I.C., F.R.S.	1944–1946	*Prof. F. M. Rowe, D.Sc., F.R.I.C., F.R.S.
1919–1938	*R. Vidal	1946–1946	*James S. Ridsdale
1921–1930	*Horace A. Lowe	1947	C. J. T. Cronshaw, D.Sc., F.R.I.C., M.I.Chem.E., F.T.I., F.R.S.E.
1923–1935	*Charles F. Cross, F.R.S.	1948	H. Jennison, M.C., A.M.C.T., A.R.I.C.
1925–1927	*Maurice Prud'homme	1950–1953	*George E. Holden, C.B.E., M.Sc., F.R.I.C.
1927–1930	*Ernest Hickson	1953	John Barritt, B.Sc., A.R.C.S., A.R.I.C.
1928–1939	*R. E. Schmidt		

The following have been *ex officio* Honorary Members of the Society, the later dates indicating termination or change in title of the office—

1886–1920	The Worshipful Master of the Dyers' Company	1886–1900	The President, Bradford Technical College
1920–	The Prime Warden of the Worshipful Company of Dyers	1900–1905	*W. E. B. Priestley, Chairman of the Technical Instruction Committee of the Bradford City Council

*Deceased

THE PERKIN MEDAL

The Perkin Medal was modelled by the late F. W. Pomeroy, R.A., for the Society. It is an excellent presentation of the head of Sir William Perkin, the founder of the coal-tar colour industry, and President of the Society in 1907.

This medal is awarded for discoveries or work of outstanding importance in connection with the tinctorial arts.

LIST OF RECIPIENTS

1908 Professors Graebe and Liebermann. "Synthesis of Alizarin."

1911 Prof. Adolf von Baeyer. "Synthesis of Indigo."

1914 Comte Hilaire de Chardonnet. "Artificial Silk."

1917 Prof. Arthur G. Green. "Primuline."

1919 R. Vidal. "Sulphur Black."

1921 Horace Lowe. "Permanent Lustre on Cotton."

1923 Chas. F. Cross. "Discovery of Viscose."

1925 M. Prud'homme. "Aniline Black and Alizarin Blue."

1928 Dr. Robert E. Schmidt. "For Epoch-making Discoveries of Anthraquinone Derivatives and Dyestuffs therefrom."

1938 Dr. H. Dreyfus. "For Discoveries and Work of Outstanding Importance in Connection with the Development of the Cellulose Acetate Rayon Industry in England."

1938 J. Baddiley. "In recognition of his National Services for the Renaissance of the British Dyestuffs Industry through Many Important Investigations in the Field of Colour Chemistry Conducted or Directed by him."

1950 Prof. J. B. Speakman. "In recognition of his Outstanding Contributions to the Science and Technology of Textiles."

THE MEDAL OF THE SOCIETY OF DYERS AND COLOURISTS

This Medal was instituted by the Society in 1908. Of the competitive designs submitted, that of Mr. Edgar Lockwood was selected. The design indicates that the work of the dyer (centre figure) is both a science (right background) and an art (left foreground).

1908–1927 The Medal was occasionally awarded as a recognition of work of exceptional merit carried out under the Society's Research Scheme.

From 1928 The Medal was awarded as a recognition of exceptional services (*a*) to the Society or (*b*) in the interests of the Tinctorial and Allied Industries.

LIST OF RECIPIENTS

1908 J. B. Fothergill (Bronze Medal). "Treatment of Cotton to Cause it to Resist Direct Dyeing Colours."

1912 J. H. Garner (Silver Medal). "Treatment of Effluents from Dyehouses and Textile Factories."

1928 Ernest Hickson (Gold Medal). "Exceptional Services rendered to the Society as Chairman of the Publications Committee 1897–1925, and Chairman of *Colour Index* Committee."

1930 Arthur Silverwood (Gold Medal). "Exceptional Services rendered to the Society as Honorary Secretary 1913–1930."

1933 Prof. Walter M. Gardner (Gold Medal). "Distinguished Services as Editor of the Society's *Journal* 1900 to 1932."

1934 Prof. F. M. Rowe (Gold Medal). "Exceptional Services to the Society and to the Tinctorial Industries as Editor of the *Colour Index* 1924 and the 'Supplement' 1928."

L. A. Lantz (*Chairman*), H. H. Bowen, P. W. Cunliffe, R. S. Horsfall, Prof. B. A. McSwiney, C. C. N. Vass, C. M. Whittaker, S. G. Barker (Silver Medals). "Exceptional Services to the Society as Members of the Standardisation of Fastness Executive Committee."

1936 W. A. Edwards (Silver Medal). "Valuable Services rendered to the Society as Honorary Secretary of the Midlands Section since its inception in 1919 to 1935."

1937 R. Ritchie (Silver Medal). "Devoted and Valuable Services as Honorary Secretary of the Scottish Section for eighteen years."

1940 F. L. Goodall (Gold Medal). "Valuable Services to the Tinctorial Industries by his work on the Theory and Practice of Wool Dyeing."

1943 C. M. Whittaker (Gold Medal). "In recognition of Exceptional Services in promoting Scientific and Technical Knowledge amongst Textile Colourists, and for Sustained and Outstanding Service to the Society."

1946 H. H. Bowen (Gold Medal). "For Outstanding Services to the Society and in recognition of his Chairmanship of the Publications Committee for a period of twenty years."

H. H. Hodgson (Gold Medal). "For Outstanding Services to the Society and for his series of Sustained Experimental and Theoretical Contributions to those chapters of Organic Chemistry which are the essential scientific background to the Dyestuffs Industry."

E. Race (Silver Medal). "For his Valued Services to the Society as Joint Author of thirteen papers published in the Society's *Journal*."

H. Turner (Silver Medal). "In recognition of his Valuable Services to the Society and of his twenty-two years' service as Honorary Secretary of the Huddersfield Section."

Mrs. E. Cummings (*née* Levin) (Silver Medal). "For her Valued Services to the Society; by her assistance rendered in the preparation of the first *Colour Index*; by her Joint Authorship of a number of Papers published in the *Journal* of the Society, and as an Abstractor for the *Journal* of the Society for twenty years."

1947 Fred Smith (Gold Medal). "For Exceptional Services to the Society over a period of thirty-seven years comprising Chairman, West Riding Section; Member of Council and of many important Committees."

F. Scholefield (Gold Medal). "In recognition of Exceptional Services to the Society in the advancement of Tinctorial Technology both in theory and practice."

C. Schardt (Silver Medal). "In recognition of his Valuable Services to the Society over a period of twenty-four years including Chairman, Vice-Chairman, and member, of the Midlands Section Committee."

1948 G. G. Hopkinson (Gold Medal). "For Valuable Services rendered to the Society and to the Dyeing Industry."

D. B. F. McAndrew (Silver Medal). "For Valuable Services rendered to the Society as Honorary Secretary and Committee Member of the Scottish Section 1935–1947."

C. O. Clark (Silver Medal). "For Valuable Services rendered to the Society from 1923 to 1948."

1949 S. M. Neale (Gold Medal). "For his Pioneer Work in the Application of the Methods of Physical Chemistry to the Elucidation of the Phenomena of Dyeing, more particularly of Cellulosic Materials with Substantive Dyes."

1950 P. W. Cunliffe (Gold Medal). "For Outstanding Services to the Society and to the Tinctorial and Allied Industries."

H. Foster (Gold Medal). "For Outstanding Services to the Society."

L. A. Lantz (Bar attached to Silver Medal previously awarded). "For Exceptional Services to the Society and to the Tinctorial and Allied Industries in connection with Fastness Tests."

1951 W. Kilby (Gold Medal). "For Work on the Development of a Molten Metal Process of Continuous Dyeing."

1953 H. A. Turner (Gold Medal). "For his Valuable Researches contributed to the Tinctorial Industries."

A. W. Carpenter (Silver Medal). "For Valuable Services to the Society."

Miss M. Forbes (Bronze Medal). "For Valuable Services to the Society."

H. R. Hadfield (Bronze Medal). "For Valuable Services to the Society."

THE WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL

The Medal represents the Arms of the Worshipful Company of Dyers of the City of London, which were granted in 1471. The following is a brief description—

Arms— Sable, a chevron engrailed argent, between three bags of madder of the last, corded or.

Crest— On a wreath three sprigs of the graintree erect vert, fructed gules.

Supporters—Two leopards rampant gardant argent, spotted with various colours; fire issuing from their ears and mouth proper, both ducally crowned or.

Motto— Da Gloriam Deo.

1— The Dyers' Company offer annually a Gold Medal called "The Worshipful Company of Dyers Research Medal", the award of which is open to the Authors of papers embodying the results of scientific research or technical investigation connected with the tinctorial arts submitted to the Society of Dyers and Colourists, and published in the *Journal* of such Society during the twelve months ending on the 30th June in the year for which the Medal is granted, and, in the special circumstance provided for by Rule 6, during the twelve months ending on the 30th June in the year previous to that for which the Medal is granted. If a paper shall be published in two or more parts, then for the purpose of the award of the Medal, all the parts together shall be treated as a paper published in the year in which the final part is published.

2— The Medal will not be awarded to the same person on more than one occasion.

3— The Society of Dyers and Colourists shall consider the papers available for the Medal and advise the Company as to the merits thereof, and if, in the judgment of the Society, none of the papers is of sufficient merit, the Society may recommend that the Medal be not awarded.

4— The Dyers' Company will award the Medal either to the Author of the paper which, in all the circumstances, appears to the Company to show the greatest merit, or, in the event of such paper being the work of an Author who has already been awarded the Medal, to the Author of the paper next in order of merit who has not already been awarded the Medal, and may, if the Company so thinks fit, refrain from making any award.

5— In the event of a paper being the work of two or more persons, the Author shall be taken to be that one of them whose work in the opinion of the Society of Dyers and Colourists has most substantially contributed to the merit of the scientific research or technical investigation embodied in such paper.

6— In the event of the Author of a paper of sufficient merit published in the *Journal* of the Society of Dyers and Colourists during any twelve months for which the Medal is awarded being unsuccessful in obtaining the award of the Medal for that period, the Society of Dyers and Colourists may, in their discretion, consider such paper for adjudication with the papers available for the award of the Medal for the next succeeding twelve months.

LIST OF RECIPIENTS

1908	Prof. E. Knecht. "A Means of Estimating the Degree of Mercerisation in Cotton Yarns."
1909	Prof. Arthur G. Green. "The Chemical Technology of Aniline Black."
1910–11	R. L. Taylor. "The Action of Carbon Dioxide and of Air on Bleaching Powder."
1911–12	W. Harrison. "The Electrical Theory of Dyeing."
1912–13	S. H. Higgins. "Observations on the Bleaching of Cotton" and "The Action of Neutral Salts on Bleaching Solutions."
1913–14	W. Johnson (in conjunction with Prof. Arthur G. Green, who had already been awarded the Medal). "The Constitution of Aged and of Bichromate Aniline Blacks."
1914–15	Morris Fort. "The Mechanism of the Acid Dye-bath."
1915–16	James R. Hannay. "The Interaction between Metallic Copper and certain Dyes of the Thiazine, Oxazine and Azine Series."
1916–17	Prof. H. M. Dawson. "The Phenomena of Acid Catalysis and the Theory of Acids."
1917–18	L. G. Radcliffe. "The Sulphonation of Fixed Oils."
1918–19	Chas. F. Cross (Diplomas presented to M. C. Lamb and C. V. Greenwood as co-authors). "Colloidal Tannin Compounds and their Applications."
1919–20	A. E. Everest (Diploma presented to A. J. Hall as co-author). "The Tinctorial Properties of some Anthocyans and certain Related Compounds."
1920–21	Prof. G. T. Morgan. "The Co-ordination Theory of Valency in Relation to Adjective Dyeing."
1921–22	S. Judd Lewis. "On the Fluorescence of Cellulose and its Derivatives."
1922–23	Prof. Arthur G. Green (Bar attached to Medal previously awarded), and K. H. Saunders. "The Ionamines— A New Class of Dyestuffs for Cellulose Acetate Silk."
1923–24	S. Judd Lewis (Bar attached to Medal previously awarded). "The Quantitative Determination of the Fluorescent Power of Cellulose and its Derivatives."
1924–25	Prof. F. M. Rowe (with Diploma to Miss E. Levin, as co-author). "The Identification of Azo Colours on the Fibre and of Azo Pigments in Substance."
1925–26	H. H. Hodgson. "Behaviour of the Sulphides of Sodium in Aqueous and Alcoholic Media", and "The Action of Sulphur on the Monochloranilines."
1926–27	Prof. F. M. Rowe (Bar attached to Medal previously awarded) and Diplomas awarded to collaborators— Miss E. Levin, A. C. Burns, J. S. H. Davies, and W. Tepper. "A New Reaction of Certain Diazosulphonates derived from β-Naphthol-1-sulphonic acid, leading to the preparation of Phthalazine, Phthalazone and Phthalimidine Derivatives."
1927–28	No award.
1928–29	F. Scholefield (with diplomas to Miss E. Hibbert and C. K. Patel as co-authors). "The Action of Light on Dyed Colours."
1929–30	H. H. Hodgson (Bar attached to Medal previously awarded). "Colour and Constitution from the Standpoint of Recent Electronic Theory."
1930–31	Prof. F. M. Rowe (Second Bar attached to Medal previously awarded), and Diplomas awarded to S. Ueno and F. H. Jowett as collaborators. "Insoluble Azo Colours on the Fibre and Action of Boiling Caustic Soda thereon."

1931–32 No award.

1932–33 Two awards— W. T. Astbury. "The X-ray Interpretation of Fibre Structure."
J. B. Speakman. "The Structure of the Wool Fibre; its Relation to the Dyeing and Finishing Processes of the Wool Textile Trade."

1933–34 No award.

1934–35 H. A. Turner, and Diplomas awarded to G. M. Nabar and F. Scholefield, as co-authors. "The effect of Reduced Vat Dyes upon the Hypochlorite Oxidation of Cellulose."

1935–36 Prof. F. M. Rowe (Third Bar attached to Medal previously awarded), and Diplomas awarded to C. H. Giles, R. L. M. Allen, W. G. Dangerfield, and Glyn Owen, as collaborators. "Decomposition of Azo Dyes by Acids, Caustic Alkalis, and Reducing Agents."

1936–37 J. B. Speakman (Bar attached to Medal previously awarded), and Diplomas awarded to C. S. Whewell and J. L. Stoves, as collaborators. "The Reactivity of the Sulphur Linkage in Animal Fibres."

1937–38 Prof. F. M. Rowe (Fourth Bar attached to Medal previously awarded), and J. B. Speakman (Second Bar attached to Medal previously awarded), and Diplomas awarded to E. Race and T. Vickerstaff, as collaborators. "The Unlevel Dyeing of Wool with Acid and Chrome Dyes. Part I— The Reasons for the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure, and Part II— A Method for Correcting the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure."

1938–39 No award.

1939–40 T. H. Morton, as senior author of the paper on "Application of Vat Dyes to Viscose Rayon", by J. Boulton and T. H. Morton.

1940–41 No award.

1941–42 T. Vickerstaff, as senior author of the paper on "The Dyeing of Cellulose Acetate Rayon with Dispersed Dyes", by T. Vickerstaff and E. Waters.

1942–43 No award.

1943–44 J. Boulton. "The Importance of Dyeing Rate— an Interpretation for the Practical Dyer of Recent Research on Direct Dyeing."

1944–45 No award.

1945–46 No award.

1946–47 H. Phillips, as senior author of the paper on "The Chemical Constitution and Physical Properties of Bisulphited Wool", by E. G. H. Carter, W. R. Middlebrook, and H. Phillips.

1947–48 J. M. Preston. "Some Factors affecting the Dyeing of Viscose" and "Some Aspects of the Drying and Heating of Textiles".

1948–49 H. Lindley, as major contributor to the paper "The Mechanism of Set and Supercontraction in Wool Fibres", by S. Blackburn and H. Lindley.

1949–50 No award.

1950–51 J. Crank. "The Diffusion of Direct Dyes into Cellulose. III— The Present State of the Theory and its Application".

1951–52 H. Hampson. "Advances in the Application of Vat Dyes to Viscose Rayon Cakes".

1952–53 R. H. Peters, as senior author of the paper on "The Reduction Properties of Vat Dyes", by W. J. Marshall and R. H. Peters.

THE WORSHIPFUL COMPANY OF FELTMAKERS RESEARCH MEDAL

The Feltmakers' Company offer annually a Gold Medal for Papers embodying the results of scientific research or technical investigation connected with the art of feltmaking and published in the *Journal* of the Society. Further, in order to encourage research of a type likely to qualify for the award of the Medal, the Feltmakers' Company offer annually, for an initial period of five years, a Research Grant of £50.

1945–1953 No award.

KNECHT MEMORIAL FUND

A Fund of £100 was subscribed as a Memorial to the late Professor Edmund Knecht, from the interest on which two prizes, in the form of books, are awarded annually to selected students of the Manchester College of Technology and the Royal Technical College, Salford.

MERCER LECTURES

A sum of £20 per annum is being given to the Society for a limited period of years in commemoration of the centenary of the discovery of mercerisation. Suitable lecturers are invited to give a Mercer Lecture annually.

1944 N. G. McCulloch and G. S. Hibbert. "Science in an Old Industry."

1945 G. S. J. White, B.A., and T. Vickerstaff, Ph.D. "Colour."

1946 D. Entwistle, B.Sc., A.R.I.C. "Regenerated Fibres from Natural Polymers."

1947 E. J. Bowen, M.A., F.R.S. "Colour and Constitution— The Absorption of Light by Chemical Compounds."

1948 F. Farrington, B.Sc., F.R.I.C. "Textile Printing."

1949 E. Wilson, B.A. "Some Applications of Chemistry to Textile Finishing."

1950 A. B. Meggy, Ph.D., A.R.I.C. "Some Recent Developments in the Theory of Dyeing."

1951 D. Traill, Ph.D., F.R.I.C., F.T.I. "Some Trials by Ingenious Inquisitive Persons— Regenerated Protein Fibres."

1952 F. C. Wood, M.Sc., Ph.D., F.R.I.C., F.T.I. "Non-felting Wool and Wool Mixtures."

1953 J. R. Blockey, M.Sc., F.R.I.C., and D. H. Tuck, A.L.C. "The Coloration of Leather."

GEORGE DOUGLAS LECTURE

A sum of £2000 was given to the Society by the Bradford Dyers' Association Ltd., the income from the investment of which is employed in providing a biennial lecture on some subject connected with the colouring or finishing of textiles.

1949 H. Levinstein, M.Sc., Ph.D., M.I.Chem.E., F.R.I.C. "George Douglas, His Times, and Some Thoughts on the Future."

1952 R. Hill, Ph.D. "Synthetic Fibres in Prospect and Retrospect."

1953 T. Holbro, Ph.D. "The Search for New Dyes in relation to Modern Developments in the Textile Field."

Proceedings of the Society

The Textile Trade and the Newer Fibres

F. H. CLAYTON

One-day Symposium on Synthetic Fibres held by the Manchester Section at the Manchester College of Technology on 13th March 1953, Mr. F. Farrington in the chair

The three main phases of growth of the textile industry are considered.

The world consumption of all fibres is still increasing, but the use of man-made fibres is increasing much more rapidly than that of the natural fibres.

The properties of the synthetic fibres are summarised and some end-uses for which they are suitable. When we know what properties are required for satisfactory service for all the various end-uses, then by skilful fabric design we shall be able to make even better textile articles than we have been used to so far.

Which is the perfect textile fibre? It does not exist, and in fact never will do so if we are looking for a single fibre which will serve every textile purpose completely and satisfactorily.

People sometimes imagine that one or another of the newer fibres will indeed be universal in the sense of being equally good for every possible use. A glance at Fig. 1 will be enough to convince you otherwise. How can we envisage one single textile fibre which could, according to need, behave as a very strong, very inextensible fibre like glass or highly oriented cellulose, or alternatively behave as a very extensible fibre like wool?

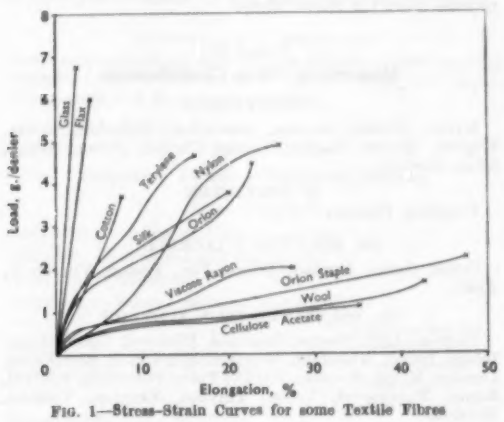


FIG. 1—Stress-Strain Curves for some Textile Fibres

Some uses or purposes require very inextensible fibres like linen or even glass, but these fibres would be useless for making, say, ladies' stockings, or knitted jumpers or socks. These garments require fabrics, and in turn fibres, which are extensible and yet which also return rapidly to their original unextended condition. With many of the properties of textile fibres, instances may be quoted where a particular fibre property is ideal for one purpose but almost useless for another.

Another question might be— Why are not the natural fibres— wool, cotton, silk, flax, jute, etc.— good enough for our needs provided they are skilfully and correctly treated or finished?

To bring the whole picture into the right perspective, we may for a moment, go back into history.

In the last three centuries there have been three distinct periods where there has been for one reason or another a major change due to invention followed by a different approach to textile fibres. Each of these periods or phases has been followed by increasingly virile growth and expansions of the whole of the textile industry.

The first phase started with the Industrial Revolution, prior to which textiles were in effect hand-made. The spinning jenny alone not only enabled yarn to be spun mechanically, in much greater quantity both total and also per man-hour than ever before, but was, in addition, responsible for the large growth in percentage consumption of cotton textiles. This was mainly because in the early stages this and other similar machines were not capable of producing woollen yarns without excessive fibre breakage. The power loom, of course, also increased fabric production tremendously.

The startling effect of the Industrial Revolution is shown in Table I. There is a most amazing reversal between wool and cotton, and yet Fig. 2 shows that the production of all these fibres went on expanding over the period from 1800 to 1950 except for a temporary decline in wool between 1800 and 1900.

TABLE I

World Production of Major Textile Fibres

	1800 %	1900 %
Wool	78	14
Cotton	4	76
Flax	18	10

About 1900 came the second phase of growth, when man found the way to make fibres from naturally occurring polymers such as the cellulose in wood, and "artificial silk" or rayon struggled into life. By 1940 rayon was no longer an infant, and had grown to real stature in the family of fibres.

From 1936 to 1940 began the third phase, which was the start of man's ability to make completely synthetic fibres, so that he had become independent even of ready-made natural polymers.

Fig. 2 shows also that the growth of these new fibres has not caused a reduction of the total consumption of cotton and wool, which, in fact, have

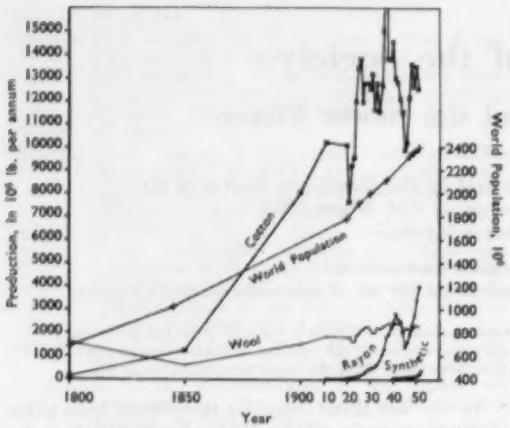
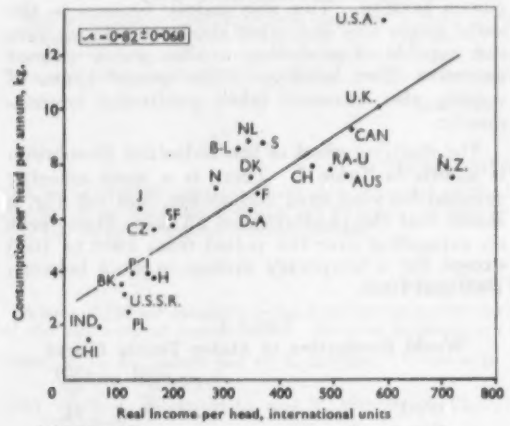


FIG. 2—Total World Fibre Production and Population Growth

both continued to expand in production. The reason for this is mainly twofold. The total world population is steadily increasing by 1% per annum, which means that there are 23 million extra people every year to clothe, but in addition, there is also a world shortage of textile fibres. Many people are short of textiles for the main reason that their standard of living is too low, as will be seen from Fig. 3, which shows the correlation of the standard of living and national textile consumption for 1938, the latest year for which we have such data.



AUS	Australia	IND	India
BK	Balkans	J	Japan
B–L	Belgium–Luxemburg	N	Norway
CAN	Canada	NL	Netherlands
CH	Switzerland	NZ	New Zealand
CHI	China	P	Portugal
CZ	Czechoslovakia	PL	Poland
D–A	Germany–Austria	RA–U	Argentina–Uruguay
DK	Denmark	S	Sweden
F	France	SF	Finland
H	Hungary	UK	United Kingdom
I	Italy	USA	United States
		USSR	Soviet Russia

FIG. 3—Relation between Real Income and Consumption per Head of Major Clothing Fibres 1934–1938 (F.A.O.)

The world average textile consumption is 8½ lb. per head of the population (or about 4 kg.—see Fig. 3). At this stage let us make a calculation of what would have happened to the total world consumption of textile fibres if certain standards of living had been raised in 1938.

Assuming that the standard of living of all those people below the average had been raised up to the average, then at least an extra 5,400 million lb. of textile fibres per annum would have been required, an extra 25%. To bring those below the world average up to half the average United Kingdom consumption at the time would have required at least an extra 17,000 million lb. per annum (= + 82%); and to bring the same people up to half the U.S.A. annual consumption would have required an extra 22,000 million lb. per annum, or more than double the world consumption of all textile fibres at that time.

So we see, that, if standards of living are going to rise for the majority of the world's population, not only shall we need more textiles in the future, but we shall also need textiles which will last longer.

Somebody might well ask how many different fibres there are at present. I do not know exactly, but excluding the natural fibres, there are at least thirty different manufactured fibres at present, with over three times that number of variants from country to country and from firm to firm, and what is more, this almost bewildering total compares with the six to eight fibres which were all that were available up to as recently as about 50 years ago.

Table II shows a convenient classification for the large array of man-made fibres, and in the different groups may be seen some of the various trade-names used for these fibres.

TABLE II

Man-made Fibre Classification

1. POLYAMIDE

Nylon, Perlon, Amilan, Austrylon, Enkalon, Grilon, Kapron, Mirlon, Nailon, Niplon, Phrilon, Polan, Rilsan, Silon, Steelon.

2. POLYESTER

Terylene, Dacron.

3A. POLYVINYL (ACRYLIC)

Orlon, dynel, Acrilan, Dolan, Pan, Redon, Vinyon N, X–51.

3B. POLYVINYL (NON-ACRYLIC)

Vinyon, Algil, Bexan, Cremona, Fibravyl, Geon, Gosei-Ichigo, Isovyl, Kanebian, Kuralon, Kurabilon, Kurehalon, Lumite, Movil, Newlon, P.C.U., PeCe, Permalon, Rhovyl, Saran, Thermovyl, Velon, Vinylan, Vinylon, Viskord, Woolon.

4. POLYETHYLENE

Polythene, Reevon, Wynene, Courlene.

5. POLYURETHANS

Perluran

6. CELLULOSIC

Cellulose acetate, cuprammonium **rayon**, viscose rayon.

7. PROTEIN

Aralac, Ardil, Cargan, Caslen, Casolana, Fibrolane, Lactofil, Lanital, Merinova, Sarelon, Silkool, soyabean, Soylon, Vicara.

8. ALGINIC

Aliginate rayon.

9. MINERAL FIBRES

Fiberglas, Fibreglass, Vitron, Fiberfrax.

10. SYNTHETIC COATED FIBRES

Plexon.

It must be emphasised that the following lists of properties (Tables III–VIII) are based on the best information available in the literature and not on the results of personal comparison or check testing.

TABLE III

Polyamide Fibres—Nylon, Perlon, etc.

Major Properties

Easy launderability and quick drying
Ability to be heat set and stabilised
Good crease retention
Resistant to insects, mildew and bacteria
Tenacity 4–7·5 g./denier } depending on whether continuous filament or staple
Elongation at break 15–35% } depending on whether continuous filament or staple
Density 1·14 g./c.c.
Softening point 480°F.
Moisture regain about 4%
Good dyeability by all methods
Moderate acid resistance
Excellent alkali resistance
High flex life and elastic recovery
Excellent resistance to abrasion

Typical End-uses

APPAREL

Stockings, underwear, dress and blouse fabrics, foundation garments, rainwear, umbrella fabrics, socks, blends with wool, rayon or cotton to give special wearing properties.

HOUSEHOLD AND INDUSTRIAL

Draperies, upholstery fabrics, pile fabrics, carpets, sewing threads, laundry fabrics, filter cloths, ropes, tyre cords, fishing nets and lines, work clothing.

TABLE IV

Polyester Fibres—Terylene, Dacron

Major Properties

Easy launderability and quick drying
Ability to be heat-set and stabilised
Good crease retention
Resistant to insects, mildew, and bacteria
Tenacity 3·5–7·5 g./denier } depending on whether continuous filament or staple
Elongation at break 7 to 35% } depending on whether continuous filament or staple
Density 1·38 g./c.c.
Softening point 480°F.
Moisture regain about 0·5%
Not easy to dye by ordinary methods
Excellent acid resistance
Moderate alkali resistance
High resistance to stretch
Good resistance to abrasion
Good resistance to sunlight (behind glass)
Good recovery from wrinkling

Typical End-uses

APPAREL

Suitings, coatings, underwear, socks, shirtings, dress and blouse fabrics.

HOUSEHOLD AND INDUSTRIAL

Curtains, upholstery, sewing thread, firehose, filter cloths, ropes, work clothing, draperies, fishing lines.

TABLE V

Polyvinyl (Non-acrylic) Fibres—Rhovyl, Vinyon, Vinylon, etc.

Major Properties

Resistant to insects, mildew, and bacteria

POLYVINYL CHLORIDE AND POLYVINYLIDENE CHLORIDE

Tenacity 2–3 g./denier
Elongation at break about 40%
Density 1·4–1·5 g./c.c.
Softening point 140–250°F.
Moisture regain 0–1%
Dyeability varies from undyeable to good
(PeCe fibre is soluble in acetone)

POLYVINYL ALCOHOL

Tenacity 2–10
Elongation at break 15–30%
Density 1·3 g./c.c.
Softening point about 250°F.
Moisture regain about 5%
Dyeability good with certain classes of dyes
High resistance to sunlight

Typical End-uses

Automobile fabrics
Filter cloths
Outdoor and indoor furniture fabrics
Screens and netting
Awnings and curtains
Luggage fabrics
Fancy braids and belts
Carpets, work clothing
Suitings, linings
Underwear, socks
Gloves and hats
Knitting yarn, sewing threads
Rainwear, umbrellas
Work clothes
Sheets and table cloths
Carpets
Filter cloths, tentage
Tyre cord, fishing nets

TABLE VI

Polyvinyl (Acrylic) Fibres—Orlon, Dynel, Acrilan, etc.

Major Properties

Easy launderability and quick drying } except for dynel*
Ability to be heat-set and stabilised } except for dynel*
Good crease retention
Resistant to insects, mildew, and bacteria
Tenacity 2–5 g./denier } depending on whether continuous filament or staple
Elongation at break 16–45% } depending on whether continuous filament or staple
Density 1·14–1·17 g./c.c.
*Softening point is 250°F. for dynel (cannot be ironed in 100% form)
Moisture regain 0·5–2%
Dyeability varies from moderately difficult to difficult
Very good acid resistance
Moderate to excellent alkali resistance
Poor resistance to abrasion
Negligible pilling
Good recovery from wrinkling
Staple has a "wool-like" handle
Filament has a "silk-like" handle
Highly resistant to sunlight and weathering

Typical End-uses

APPAREL

Outerwear— both men's and women's suitings, etc.
Underwear and nightwear
Shirtings, socks
Overcoatings, rainwear
Sportswear, neckwear
Umbrella fabrics
Blends with wool, rayon, and cotton
Decorative resist stripes in fabrics made from other fibres

HOUSEHOLD AND INDUSTRIAL

Curtains, window shades, awnings, draperies, carpets, blankets, motor car hoods, tents, tarpaulins, outdoor furniture, sail cloth, filter fabrics, electrical insulation, fishing nets, sewing threads, hosiery dye nets, anode bags, work clothing

TABLE VII

Polyethylene Fibres— Courlene, Polythene, etc.

Major Properties

Resistant to insects, mildew, and bacteria
Density 0·92 g./c.c.— the only known solid fibre lighter than water
Softening point 170–220°F.
Moisture regain zero
Cannot be dyed, but is coloured before melt extrusion
Resistant to most chemicals, though affected by toluene, hydrocarbons, and chlorinated solvents at elevated temperature
Good resistance to abrasion
Good resistance to sunlight and weathering, remains inherently flexible at very low temperatures
Excellent frequency insulation
Waxy handle

Typical End-uses

Automobile fabrics
Outdoor and indoor furniture seating fabrics
Luggage fabrics
Collar interlinings for fusing
Women's hats, handbags, and shoe fabrics
Fancy braids and bolts

TABLE VIII

Regenerated Protein Fibres— Ardil, Lanital, Fibrolane, Vicara, etc.

Major Properties

Resistant to insects, mildew, and bacteria
Tenacity 0·5–1·2 g./denier (dry) (low wet strength)
Elongation at break 35–50%
Density 1·25–1·3 g./c.c.
Moisture regain 12–13%
Good dyeability generally
Moderate resistance to acids and alkalis
Poor resistance to abrasion
Will not itself shrink or felt like wool, but assists the felting of wool in blends
Soft, warm, and fairly resilient handle
Colour ranges from cream to fawn
Generally, but not in all cases unsuitable for spinning as 100% yarn

Typical End-uses

APPAREL WEAR

Suitings, dress fabrics, half-hose, underwear, knitted outerwear, hand knitting yarns, pile fabrics

HOUSEHOLD

Blankets, upholstery, carpets, felts

It is not always easy to make a strict comparison of the properties of various different fibres, and it is only as a result of extensive trial that the virtues and the snags show themselves in their true light, so that one can speak with much more confidence about the behaviour of those fibres which have stood up to several years of exhaustive trial and end-use performance.

To be successful, a textile fibre must have the following merits in descending order of importance—

(i) Sufficient outstanding properties of merit to ensure adequate demand in at least one major consumer market
(ii) The right price
(iii) The minimum number of shortcomings, e.g. very low melting point or major dyeing problems.

If we take only the price factor, it is quite possible to produce an outstanding new fibre for some particular purpose or other but at an exorbitant selling price. The right price is a "must": if jute sold at the present price of silk, at about 400d. instead of at about 9d. per lb., the world consumption of jute would not be at the present rate of about 3000 million lb. per annum.

A rough idea of present prices can be obtained from Table IX. Any firm attempting to produce a new synthetic fibre will have to consider this factor very carefully indeed, because these fibres cost vast sums of money before they even get to the plant-production stage, and to be competitive they must have distinctive, useful characteristics.

TABLE IX

Comparative Prices of Textile Fibres

(Prices in pence per lb. in April 1952)
(Prices are those for the U.K. except where stated otherwise)

			d.
	SYNTHETIC		
	Nylon	60 denier	179
	Nylon	Staple	135
	Terylene	70 denier	219
(U.S.A.)	Dacron	70 denier	198
(U.S.A.)	Dacron	Staple	155
(U.S.A.)	Orlon	75 denier	322
(U.S.A.)	Orlon	Staple	163
(U.S.A.)	Dynel		120
(France)	Rhovyl		157
(France)	Fibravyl		101
(Japan)	Vinylon		80?
	Fibreglass	900/1/0	149
	Fibreglass	Staple	72
	NATURAL		
	Cotton	(Amer. Midd.)	41
	Wool	(64s tops)	122
	Flax	(Grade DD)	44
	Silk	(20/22–90%)	436
	REGENERATED		
	Viscose rayon	75 denier	70
	Viscose rayon	Staple	27
	Acetate rayon	75 denier	70
	Acetate rayon	Staple	41
	Cuprammonium rayon	60 denier	91
	Ardil		50
(U.S.A.)	Vicara		86

TABLE X

Price Stability of Manufactured Fibres

(Prices are for fibre sold in the U.S.A., but are converted into sterling—pence per lb.)

	1948	1949	1950	1951	To July 1952
NATURAL FIBRES					
Cotton	27–32	25–28	26–36	30–37	31–36
Wool	106–156	118–165	126–223	141–308	117–148
Silk	223	300	244–380	347–510	405–468
MANUFACTURED FIBRES					
Filament					
Viscose rayon 150 denier	64	63	67	67	67
Acetate rayon 150 denier	63	62	63	65	60
Nylon 15 denier	472	515	515	515	515
Nylon 70 denier	150	163	163	163	163
Orlon 75 denier	—	—	322	322	322
Dacron 70 denier	—	—	176	202	202
Staple					
Viscose rayon 1·5 denier	32	31	33	35	35
Acetate rayon 3 denier	41	37	36	42	38
Nylon 1·5 denier	—	150	150	150	150
Dynel	—	107	107	107	110
Vicara	—	71	71	86	86
Orlon	—	—	163	163	163
Dacron	—	—	154	154	154
Vinyon	—	—	73	77	77
Acrilan	—	—	—	159	159

Another important factor is price stability, and Table X shows how little the prices of the man-made fibres have varied over the past five years, as compared with the price variations of the natural fibres.

The American attitude to new ideas is something which will act as a fairly rapid sorting house for the good and the not so good, both in performance and in value, so we should keep a careful check on textile events in that country, because the three basic ideas of less work (needed in laundering, etc.), longer life (say less darning, etc.) and better value for money, are what we here are all going to need if we are to hold our position in the world of textiles.

Table XI gives the present world production and future planned production of the various synthetic fibres. Even in total this does not yet make a big contribution. What is important, however, is the rate of growth of synthetic fibres, which may be compared with the early growth of rayon fibres (Fig. 4).

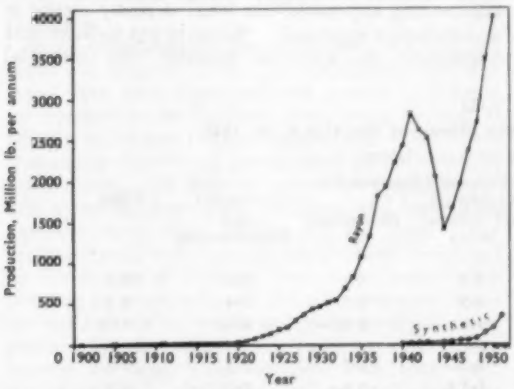


FIG. 4—Comparison of Growth of Rayon and Synthetic Fibres

TABLE XI

Planned World Production of Synthetic Fibres

Fibres	1952	1955 (Estimated)
	(Million lb. per annum)	
POLYAMIDES		
Nylon, Perlon, etc. ...	210	430
POLYESTERS		
Terylene and Dacron ...	5	46
POLYVINYL (ACRYLIC)		
Orlon, dynel, Acrilan, etc. ...	43	104
POLYVINYL (NON-ACRYLIC)		
Rhovyl, P.C.U., Saran, Vinylon, etc.	26	46
POLYETHYLENE		
Courlene, Polythene, Reevon, Wynene etc.	2	4

The difference in growth is phenomenal. The present world production of acetate, cuprammonium, and viscose rayons is about 4000 million lb. per annum, or nearly 70% greater than the present total world production of clean wool. It is also interesting to see that it has taken only 12 years for the production of synthetic fibres to reach the same amount as was attained by the rayon fibres 30–35 years from the start.

Some recent data from the U.S.A. on the total consumption per capita of the various kinds of fibres are especially interesting in connection with the previous growth chart comparison. There is, however, a warning necessary here—the figures are for the U.S.A. (they do not seem to be available for this country), and the total consumption per head of all fibres in the U.S.A. is now about 45 lb. per annum, which is the highest for any country in the world. In this country it is only about 37·5 lb. per annum.

TABLE XII

Relative Fibre Consumption in the U.S.A.

(% of total consumption)

	1930	1940	1950	1951	1952 (July)	1953 (Estimated)
Cotton ...	85	80	69	72	73	70
Wool	9	8	10	7	7	6
Silk	3	3	—	—	—	—
Manufactured total	3	9	21	21	20	24
Regenerated	3	9	19	18	16	18
Synthetic ...	0	0	2	3	4	6

There are snags with the synthetic fibres—some will not dye, some melt at low temperatures, some even dissolve in soap and water, but the difficulties can often be utilised and made their most important property. For example, sodium alginate fibre dissolves in soap and water, but it is ideal for use as a supporting thread in the dry state and a vanishing thread when wet, such as for the joining threads for continuous sock production, etc.

In this country we have a lot of textile-production equipment which is suited only to handling staple fibres. The impression might be gained that, since Lancashire is traditionally a cotton-manufacturing county, then because of the growth of synthetic fibres, its total textile output will fall in the future. This does not follow at all. The total amount of textiles being produced in the world is rising. It is quite true that, when other countries not so equipped wish to commence textile manufacturing themselves, they almost invariably start with cotton, because after all, it is usually easily the biggest item in each country's textile-importing budget. It is most important to realise that synthetic fibres, just like the rayon fibres before them, will not displace the textile workers of this country. The same workers, whether they are spinners, weavers, finishers, or makers-up of garments, can handle the synthetic fibres. What is more, it is better for our country, because with our need to maintain our export trade, it pays us much better to produce high-quality textiles which other people cannot handle so well. Also, it is undoubtedly true to say that there are still some problems with some of the newer fibres. What is even more important to Britain is that synthetic fibres produced in this country do not use up our precious foreign currency and yet we can reap the benefits on the exports.

Some people in the textile industry have looked askance at these new fibres and, in fact, are antagonistic to them. What is the real position?

There is no real battle of the fibres, and talking about such an idea only divides the industry and confuses its customers. What is actually happening is the greatest revolution in the industry since the Industrial Revolution. In fact, it will probably in future be referred to as the "textile revolution".

Up to a few years ago in this country one thought only in terms of the cotton industry, the wool industry, the silk industry, and so on. Furthermore, a firm in, say, the wool industry never handled anything at all but wool. They had never even thought of doing so, and what is more were not inclined to try.

Even today, and the new revolution has only just started, it is already becoming obvious that a cotton firm can and does handle more than cotton. Some silk spinners are actually processing up to five different textile fibres on their equipment, and so for the first time in history, we are dropping the barriers and hurdles which divided cotton from wool, the wool industry from the silk industry, and so on, and now can talk and think and work for the first time in the textile industry. What is the cause of this revolutionary change? Surely it is the fact that synthetic fibres and also the rayon fibres before them can be made suitable for processing on any and every type of textile machinery. A sample of cloth containing 25% cotton, 25% viscose rayon, 30% acetate rayon, and 20% nylon and another with 60% wool, 15% viscose rayon, 15% nylon, and 10% acetate rayon are evidence of a real revolution in outlook.

That is the essential benefit which these fibres can now offer to textiles, and yet some people still think it unwise to make any fabric containing more than one fibre. Now the synthetic fibre which is produced in largest quantity is nylon, and yet if all the nylon in the world, both continuous filament and staple, were blended with all the wool, then the blend would be only 91% wool and 9% nylon. In fact, this would produce a very good product which would be good for wear and good customer value.

All this is leading up to an entirely new approach to textiles, which might almost be termed an engineering approach, but what is really meant is an intelligent approach. We have got to learn and appreciate as soon as possible the essential

TABLE XIII

Consumption per capita of Textile Fibres in the U.S.A. in 1949

(lb. per head of the total population)

Textile Fibre	End-use Classification: Men's and Boys' Wear	Women's and Misses' Wear	Children's and Infants' Wear	Household	Industrial and Miscellaneous	Total
NATURAL						
Cotton	6·0	2·1	1·3	6·5	7·9	23·8
Wool	1·3	1·3	0·3	1·5	0·4	4·8
Silk and linen ...	0·03	0·03	—	0·01—	0·01	0·08
MANUFACTURED						
Total	0·9	2·7	0·2—	0·5	2·3	6·6
Filament	0·4+	2·2	0·1+	0·4—	2·1	5·3
Staple...	0·4+	0·5	0·02	0·2—	0·2	1·3

properties of all the textile fibres available—this may be hard, but it is easy compared with finding out the essential requirements for service in the whole gamut of end-uses for textiles, which is much more important.

What are the properties we want in a shirting cloth or a tarpaulin, in women's lingerie, or in a haulage rope, and so on? When we know what we want—and it is surprising how little we know about these things—then we can proceed to pick out the one fibre, or the blend of fibres, which will give the requisite characteristics for success, and what is more, success in the complete finished article ready for use. It is no use with a nice handle if it is going to melt on ironing, and it is no use making a garment from fabric which is tough and strong if the sewing thread will not stand up to a quarter of the life of the garment.

The idea of blends is not a new one. It is very important to know how to set and finish fabrics made from synthetic fibres, especially in blends. We do not know all the answers yet, but we do know that the property of being set is one of the most important possessed by some of the synthetic fibres. The skilled and successful manufacturer will see to it not only that the resulting cloth is satisfactorily set and stabilised for use, but also that the blend is such that it will behave satisfactorily with normal and reasonable performance in that end-use.

What is all this effort devoted to? Neither more nor less than giving the ultimate customer—the user of the commodity—the best possible article at the most economic price.

When we have all had a better acquaintance with synthetic fibres, we will change our standards a lot, and no longer make mental comparisons with the natural fibres. For example, it is now well understood that for wear life and abrasion resistance the best fibre of all is nylon. So what we must look forward to is what we shall think about these new fibres when everybody in the textile trade has contributed their share to making the best possible article for the purpose.

BRITISH NYLON SPINNERS LTD.
PONTYPOOL
MONMOUTHSHIRE

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Studies in the Printing of Terylene Polyester Fibre

A. G. H. MICHIE

One-day Symposium on Synthetic Fibres held by the Manchester Section at the Manchester College of Technology on 13th March 1953, Mr. F. Farrington in the chair

Experimental work has shown that the Dispersol and Duranol dyes, Alcian Blue 8G, pigments in the form of pigment printing compositions, and vat dyes may be applied to Terylene fibre by printing techniques. The methods of application are outlined with stress being placed on the use of pressure steaming and swelling agents for the disperse dyes. Vat dyes are applied by the acid–leuco technique and require to be steamed at high pressures for satisfactory fixation. Their light fastness when printed on Terylene fibre is lower in practically all cases compared with prints on cotton. The mechanical conditions necessary to obtain prints of satisfactory quality on Terylene fibre are discussed.

The experimental work which has been carried out on the printing of Terylene fibre has resulted in application techniques being developed for the following types of dye—

(1) Duranol and Dispersol dyes

(2) Alcian Blue 8G

(3) Vat dyes

(4) Pigments using pigment printing compositions.

Some comments on the application of these dye types will be given later. All the other known dye types have been examined, but none is of interest for application to Terylene fibre owing to lack of affinity or inferior fastness properties. Thus, dyes containing solubilising groups such as the Soledon, acid, direct, and Solacet types have insufficient affinity to be of interest. Sulphur and basic dyes likewise show little affinity for the fibre. The modified azoic technique, which has been developed for the dyeing of Terylene fibre, is not suitable for printing as practically all the recommended bases are too volatile in steam and yield poorly defined prints. The few satisfactory bases have very poor fastness to light when coupled with beta oxynaphthoic acid. Oxidation colours fail similarly owing to the volatility of the bases. It has not been found possible to introduce metallic salts into Terylene fibre and therefore mordant dyes are not of interest. Selected water-insoluble dyes from the Waxoline and Monolite classes are applicable, but the light fastness properties are generally poor.

It is necessary at this point to examine briefly the work of Waters[1] on the dyeing characteristics of Terylene fibre. He established that, even with the preferred Duranol and Dispersol dyes, Terylene fibre was not as readily dyed as acetate rayon or nylon, and he set out to determine whether this was due to Terylene fibre being incapable of absorbing sufficient dye or whether the dye had difficulty in entering and diffusing into the fibre.

In order to do this the diffusion coefficients and the saturation values of a number of dispersed dyes were measured in Terylene fibre. The results obtained from this work clearly established two facts as follows—

(1) Terylene fibre will ultimately absorb more dye than nylon and only slightly less than acetate rayon.

(2) The relative diffusion coefficients within Terylene fibre, acetate rayon, and nylon at 85°C. are approximately as 1 : 500 : 700.

Thus, the dominant factor in the coloration of Terylene fibre was the very slow rate at which dyes diffuse into the fibre. From the dye-users' viewpoint, this means that the conditions of application must be adjusted in such a manner that the diffusion of the dye into the fibre is facilitated to the maximum possible extent.

Considering steaming conditions, therefore, it has been established that even with the most readily applied Duranol and Dispersol dyes prolonged steaming is necessary under atmospheric pressure conditions in order that acceptable depths of shade may be realised. By utilising the higher temperatures available when steaming is carried out under superatmospheric pressure conditions, the rate of dye diffusion is markedly increased and it is possible to use progressively decreasing steaming times as the pressure is raised. In addition, certain types of dye, such as the vat dyes, which yield only the palest of shades under atmospheric pressure steaming conditions, may be applied by steaming at pressures of 20–40 lb./sq. in.

As an alternative to steaming under pressure the use of swelling agents has been examined. These agents open up the internal structure of the fibre and enable the dyes to diffuse in more rapidly. A wide range of potential swelling agents has been examined for use under printing conditions and the most effective agent is *p*-hydroxydiphenyl. The effect of these agents is greatest under atmospheric pressure steaming conditions and as the steaming pressure is raised the advantage gained from their employment decreases. If a steaming pressure of 10 lb./sq. in. is available then there is only a slight increase in the dye absorbed in the presence of a swelling agent, though the time taken

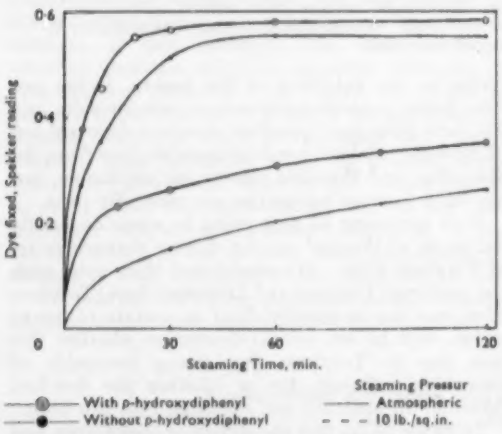

FIG. 1— 10% Duranol Red X3B 200 Paste Fine printed on Terylene Polyester Fibre

to reach equilibrium is shortened. Fig. 1 shows the dye fixation achieved by printing 10% Duranol Red X3B 200 Paste fine on to Terylene fabric, with and without the addition of *p*-hydroxydiphenyl, and steaming at atmospheric pressure and 10 lb. per sq. in. pressure for times up to 120 min. The steamed prints were given a light soap at 60°c. and standard sized patterns cut from the printed stripes. The dye present in the patterns was extracted by heating in monochlorobenzene at 95°c. After cooling and making up to volume the solutions were measured on the Hilger Spekker photoelectric absorptiometer.

Turning to the dyes suitable for application to Terylene fibre, there are firstly the Duranol and Dispersol dyes. The method of application is similar to that used for applying these dyes to acetate rayon with the swelling agent added if required. As a rough guide it may be stated that on steaming prints of the more suitable Duranol and Dispersol dyes on Terylene fibre for 30 min. at 10 lb./sq. in. pressure equal depths of shade are obtained to those realised on acetate rayon steamed at atmospheric pressure for the same time.

The washing fastness of the Duranol and Dispersol dyes on Terylene fibre is superior to that on acetate rayon or nylon. This is particularly true for the more completely penetrated prints obtained by steaming under pressure, which possess acceptable standards of fastness to repeated severe washing. The light fastness of these dyes is generally not high, mainly owing to the fact that many show marked changes in shade when exposed to light. However, by selecting the dyes, a wide range of shades may be obtained with a lower light fastness limit of 4–5. When printing Terylene fibre it is still necessary to avoid those dyes which sublime in steam, especially as any staining which occurs will be removed only with difficulty.

The application of pigment printing compositions to Terylene fibre enables it to be coloured by a conventional technique without any modification whatsoever. Under the best conditions pigment printing compositions yield prints, having good to very good fastness to washing and excellent fastness to light, though the fastness to rubbing is generally not good. Particularly close attention must be paid towards selecting the correct mechanical conditions in order to obtain smooth prints but the addition of a suitable wetting agent may prove of limited assistance.

Alcian Blue 8G is preferably applied to Terylene fibre by the acetic acid–sodium acetate method and fixed with or without pressure steaming. Despite the fact that very little penetration of the fibre is obtained the fastness to washing and rubbing are good. The light fastness is the same as that on cotton.

Intensive efforts have been directed towards the application of vat dyes to Terylene fibre by printing techniques. This effort was justified as it was hoped that very high fastness standards would be obtained, particularly fastness to light, commensurate with the anticipated life of the fibre.

The preliminary experiments indicate that the alkaline leuco form or the unreduced dye had very little affinity for the fibre and that the most promising method of application involved the use of the acid leuco compound*.

The production of the acid leuco compound is possible by two methods—

(1) By reducing the thickened vat dye in the presence of a dispersing agent but without the

* Patent protection pending

addition of alkali. The preferred reducing agent for this process is zinc formaldehyde-sulphoxylate, and the acid leuco derivative of the vat dye is producing during steaming.

(2) By pre-reducing the vat dye with caustic soda and sodium hydrosulphite and then acidifying with acetic acid in the presence of a dispersing agent. Thickening is then added together with zinc formaldehyde-sulphoxylate, the latter being required to ensure reduction of any dye which is oxidised during the drying of the prints.

There is little to choose between the results obtained with the two methods, but the former was used almost exclusively on the grounds of simplicity.

Even with this preferred method of application steaming at a pressure of 20 lb./sq. in. upwards was necessary to obtain full shades with selected dyestuffs. Microscopical examination of cross-sections showed that at 20 lb./sq. in. some, but not complete, penetration of the fibre had been achieved after 30 min. and that at 40 lb./sq. in. penetration was complete. Visually, much heavier shades were obtained at 40 compared with those obtained at 20 lb./sq. in.

Oxidation of the dyes was most readily achieved with nitrous acid at 50°C., but this destroyed dyes such as Caledon Blue GCP. When this and similar dyes were present, oxidation in ammoniacal hydrogen peroxide at the boil is employed.

Very recent work has resulted in the development of an interesting modification of the pad steam method. The print paste employed in this instance contained only dyestuff and dispersing agent so that it was now possible to use a sodium alginate thickening. These prints could be stored prior to steaming which was preceded by padding through a zinc formaldehyde-sulphoxylate solution, which precipitated the thickening onto the fabric, and drying. After oxidation finishing of the prints was completed by soaping at the boil.

Unfortunately, although prints of very high fastness to wet processing were obtained by this process, the light fastness of practically all the vat dyes was much lower than that normally associated with this type of dye. Thus, despite the very severe application conditions, vat dyes do not show very marked advantages over selected disperse dyes which can be fixed more readily.

The choice of thickening agent and the adjustment of the mechanical conditions in both screen and roller printing are influenced mainly by the low moisture absorbency of Terylene fabrics.

The operation of screen printing Terylene fabrics should not present major difficulties. The printing conditions should be adjusted to ensure that the minimum amount of printing paste is applied to the fabric consistent with adequate coverage. In general high solid content thickenings yield the sharpest printing mark but considerable care must be exercised in handling the printed goods after drying in order to avoid cracking of the printed film. Of the low solid content thickenings sodium alginate has proved most satisfactory, giving pliable printed films of good adhesion.

In roller printing shallow engravings are essential and high solid content thickenings recommended to maintain the sharpest printing mark. Nafka Crystal Gum has proved superior to gum senegal in giving more complete transfer of dye without discolouring Terylene fibre unless very high steaming pressures are employed. British Gum thickenings do not discolour Terylene and give good colour yields, but the definition is inferior and they possess poor adhesion. Sodium alginate thickenings are likely to prove the most interesting of the low solid content type.

The mechanical conditions need very careful adjustment on the printing machine to ensure a smooth and even print irrespective of thickening agent and backgreys are generally necessary. Some assistance both in facilitating wetting of the material and preventing frothing is obtained by adding Perminal KB to the printing paste.

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(*MS. received 2nd April 1953*)

Reference

[1] Waters, E., *J.S.D.C.*, **66**, 609 (1950).

Discussion

Mr. F. CROMPTON: Does Terylene have a reducing action on vat dyes as nylon does?

Mr. MICHIE: No, I am not aware of any evidence indicating that Terylene exerts a reducing action on vat dyestuffs.

Mr. R. FLETCHER: Has Mr. Michie any experience in securing Terylene to a screen printing table? Using natural gums I can assure Mr. Michie that it is a most difficult proposition.

Mr. MICHIE: I suggest that some of the synthetic resin type screen table adhesives would provide the answer or, alternatively, bonding the Terylene fabric onto a cotton backgrey which is then stuck down onto the screen table.

Mr. C. P. TATTERSFIELD: Can the lecturer say anything about discharge prints on Terylene.

Mr. MICHIE: Work on discharge styles is progressing, but I regret that at the moment I am unable to make any recommendations.

The Heat Setting of Terylene Polyester Filament Fabrics in Relation to Dyeing and Finishing

D. N. MARVIN

One-day Symposium on Synthetic Fibres held by the Manchester Section at the Manchester College of Technology on 13th March 1953, Mr. F. Farrington in the chair

The dry heat setting of Terylene filament fabrics is considered in relation to dimensional stability, wet creasing, stiffness and recovery from dry creasing, and dyeing properties. The effects of aqueous heat are also discussed. Setting machines are described, and the setting conditions for Terylene fabrics outlined.

INTRODUCTION

Terylene polyester filament yarn is derived from the polymer polyethylene terephthalate, which is melt-spun. The molten polymer, which is pumped through a spinneret, solidifies on emerging into the air and is collected on a bobbin. In this form the yarn is unoriented and lacks any textile qualities; these, however, are developed by a drawing process, which orients the yarn and gives a yarn of high strength.

Arising from this method of manufacture, Terylene filament yarn and fabrics made from it possess the property of shrinking or retracting when exposed to elevated temperatures; thus, Terylene yarn, in an unrestricted state, shrinks in boiling water about 7%. The higher the temperature to which the yarn or fabric is exposed, the higher the resultant shrinkage, and an approximately linear relationship exists between shrinkage and temperature over the range 100–200°C. (Fig. 1).

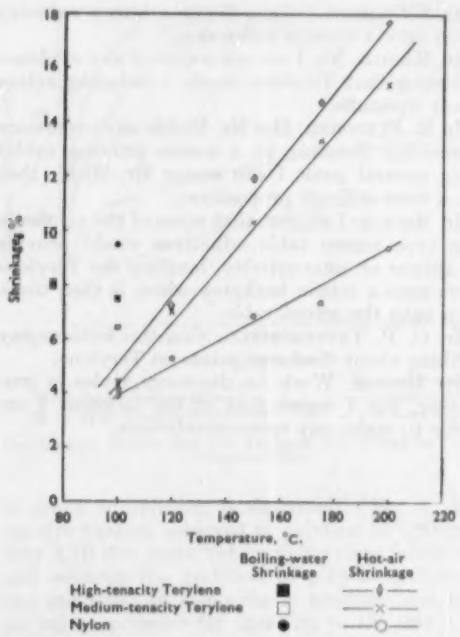

FIG. 1—Shrinkage of Terylene and Nylon Yarns in Hot Air and Boiling Water

In view of this property of Terylene fabrics it is clearly necessary, during finishing, to confer some degree of dimensional stability on them if they and garments made from them are to retain their shape on being subjected to such operations as washing and ironing. For many industrial applications, also, Terylene fabrics have to be dimensionally stable at elevated temperatures. For example, in the varnishing of electrical fabrics and the polyvinyl chloride coating of fabrics, curing temperatures of 150°C. and 180°C. respectively are encountered.

The process of stabilisation, or heat setting, as it is termed, consists essentially in exposing the fabric, while under dimensional control, to a temperature higher than any likely to be met with in its subsequent history.

Heat-setting machines available in the finishing industry may be classified into two main groups—those which use dry heat for raising the fabric to the required setting temperature and those which use aqueous heat. The heat setting of Terylene fabrics using both forms of heating will now be considered.

I. THE USE OF DRY HEAT

The effect, on the dimensional stability and other properties of Terylene filament fabrics, of setting in air and/or gases at temperatures of 120–220°C. has been determined both in the laboratory and on commercial hot-air pin-stenter setting machines with good agreement of results. The dry heat setting of Terylene fabrics in air and/or gas will be considered in relation to its effect on—

(a) *Dimensional Stability*

A graphical interpretation of the results (Fig. 2) indicates the effect of setting temperature on fabric dimensional stability. The shrinkage figures plotted were obtained from laboratory experiments in which scoured pieces of fabric were set at fixed length. They represent the linear shrinkages which occurred warpwise and which were slightly higher than the corresponding weftwise figures. A family of curves is obtained representing the various setting temperatures, and these show that, in general, the higher the setting temperature, the lower the shrinkage of the fabric at any given shrinkage temperature. Thus, at 175°C. the shrinkages of the unset fabric and scoured fabrics

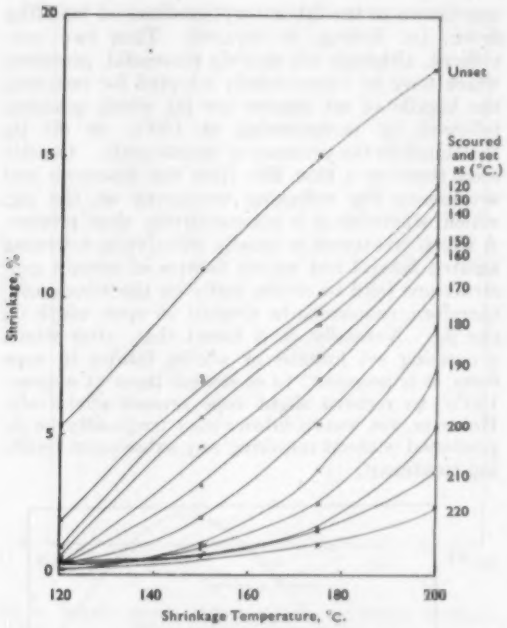

FIG. 2—Effect of Setting Temperature on Dimensional Stability of Fabrics

set at 120, 170, and 220°C. are respectively 15, 10, 5·5, and 1%. The shrinkage of all the set fabrics in boiling water is less than 2%, but this is because the fabric was free to shrink in the initial scouring process, prior to setting.

Further points which are revealed more clearly in Fig. 3 show that if, for example, a fabric is required to be made dimensionally stable at temperatures up to and including 150°C., this can be achieved by setting the fabric at a temperature of 180°C. and no significant reduction in the shrinkage of the fabric at 150°C., approx. 1%, is obtained even if it is set at higher temperatures, say 220°C. Similarly, a fabric may be stabilised at 175°C. if it is set at a temperature of 200°C. and no significant improvement in the shrinkage properties, namely 1·5%, is obtained by setting at a higher temperature than this.

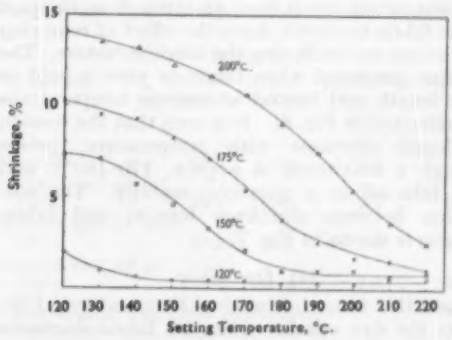

FIG. 3—Effect of Setting Temperature on Dimensional Stability of Fabric

On the basis of these results, it is, therefore, possible to generalise and say that a fabric will be effectively dimensionally stable if it is set at a temperature 30 to 40°C. higher than that at which it is required to be stable, and furthermore, no significant advantage in stability is to be gained by setting at a temperature higher than this.

Dimensional stability, i.e. the linear shrinkage which occurs warpwise and weftwise, was determined by measuring the dimensional change of fabric exposed for 15 min., in an unrestrained state, at the required shrinkage temperature. This shrinkage time was considered adequate since the majority of the shrinkage occurred well within this period and it was found that only a further 0·25% shrinkage occurred as a result of increasing the time to 45 minutes.

(*b*) *Wet Creasing*

The effect of setting a fabric is to decrease the amount by which it creases on boiling, and furthermore, the higher the setting temperature the less the creasing. This was demonstrated on an unset fabric and fabrics set at temperatures from 100° to 220°C. by boiling them in water in a crushed state for one hour. The unset fabric contained more and, incidentally, heavier creases than the set fabrics which showed a decrease in the number and severity of creases with increasing setting temperature. Whereas the creases in the set fabric were readily removable on ironing, those in the unset fabric were very difficult to remove. This was because they had been "set in" in the latter fabric.

Similarly it was found that on handwashing the fabrics for 15 minutes is a solution held at 45°C. and containing Lissapol N (2 g./litre) and soda ash, the degree of creasing decreased with increasing setting temperature. Furthermore, the ease with which the creases could be removed by ironing increased with increasing setting temperature and was most difficult in the case of the unset fabric. The creases formed on handwashing are slight by comparison with those which result on boiling.

Incidentally it should be noted at this juncture that the degree of creasing on handwashing or boiling is also related to fabric construction. Fabrics tend to crease on washing in a Bendix machine or hydroextracting, the degree of creasing depending on fabric construction, and this is caused by centrifugal loading of the fabric in a crushed state. The creases are less marked the higher the setting temperature and may be readily removed by ironing at 130–140°C. or Hoffman pressing at 30–40/lb. sq. in. steam pressure.

(*c*) *Stiffness and Recovery from Dry Creasing*

The setting of Terylene fabric is accompanied by a harshening or stiffening of the fabric which detracts from its handle and draping qualities. Measurements of the bending length, i.e. stiffness, of set fabrics have shown that their stiffness increases with increasing setting temperature and a fair linear correlation exists between these two parameters (Fig. 4). In the case of one particular fabric the stiffness increased from a value of 2·2 on

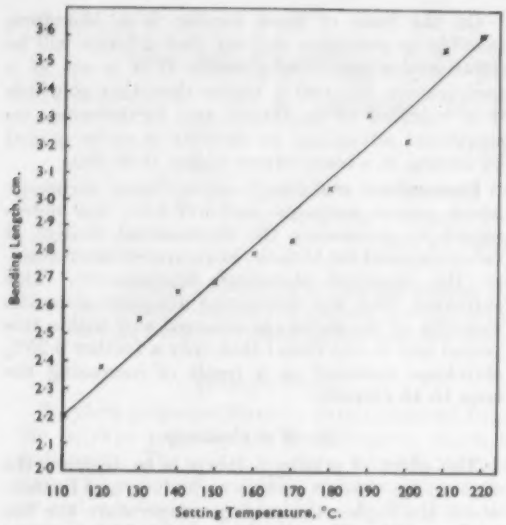

FIG. 4— Effect of Setting Temperature on Fabric Stiffness

the scoured unset fabric to 3·6 on the fabric set at 220°C.

Measurements of crease recovery have revealed that the power of recovery of a set fabric from dry creasing decreases with increasing setting temperature, although the effect is not marked until a setting temperature of 170°C. is exceeded (Fig. 5).

It has been shown in the laboratory that the handle and the crease-recovery properties of fabrics set at temperatures up to and including 220°C. may be restored by as simple a mechanical treatment as handwashing and some effect is obtained merely on dry flexing by hand. Thus, the stiffening effect which occurs when Terylene fabrics are set is simply an ephemeral condition which is most probably due to a geometrical property of the fabric and constituent yarns and not to an inherent property of the set fibre.

With this knowledge, experiments have been made to ascertain the most effective finishing equipment for breaking down the stiff handle of set fabrics and these have shown that for this purpose, a winch is more effective than a jig stenter which in turn is more effective than a jig. Calendering, as would be expected, has no effect, since, as

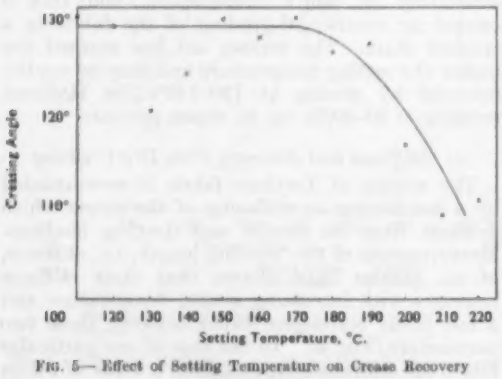

FIG. 5— Effect of Setting Temperature on Crease Recovery

was shown in the laboratory, mechanical breaking down, i.e. flexing, is required. Thus two convenient, although not entirely successful, processes which may be commercially adopted for restoring the handle of set fabrics are (*a*) winch scouring followed by re-stentering at 150°C. or (*b*) jig treatment in the presence of caustic soda. Caustic soda removes a thin film from the filaments and accelerates the softening treatment on the jig, which otherwise is a comparatively slow process. A winch treatment is usually effective in softening knitted fabrics but woven fabrics of certain constructions tend to crease badly on the winch and, therefore, require to be treated at open width on the jig. Normally, it is found that, after winch processing set knitted or woven fabrics in rope form, it is necessary to re-stenter them at approx. 150°C. to remove slight rope creases effectively. However, set woven fabrics may frequently be jig processed without requiring any subsequent finishing treatment.

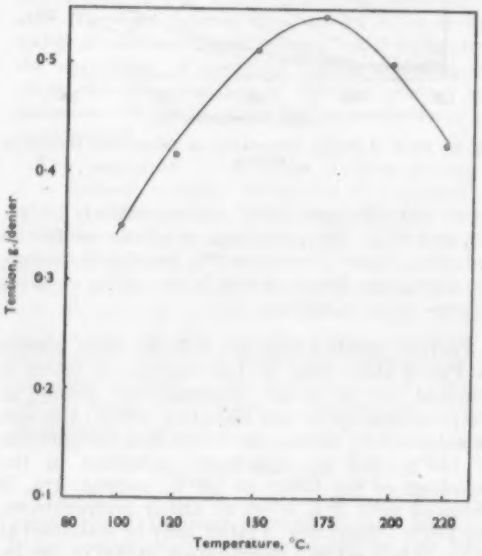

FIG. 6— Heat Setting of Terylene— Tension at Constant Length after 1 min. Exposure

The stiffening of Terylene fabrics is due to the high tensions developed during setting. These tensions, which result from an attempt on the part of the fabric to shrink, have the effect of removing yarn crimp and stiffening the fabric structure. The tensions generated when fabric or yarn is held at fixed length and heated at various temperatures are indicated in Fig. 6. It is seen that the tension developed increases with temperature, passes through a maximum at approx. 170–180°C. and then falls off in a parabolic manner. The correlation between shrinkage tension and fabric stiffness is shown in Fig. 7.

(*d*) *Dyeability*

Over the range of setting temperatures 120–230°C. the dye uptake of the set fabric decreases with increasing setting temperature, and except at

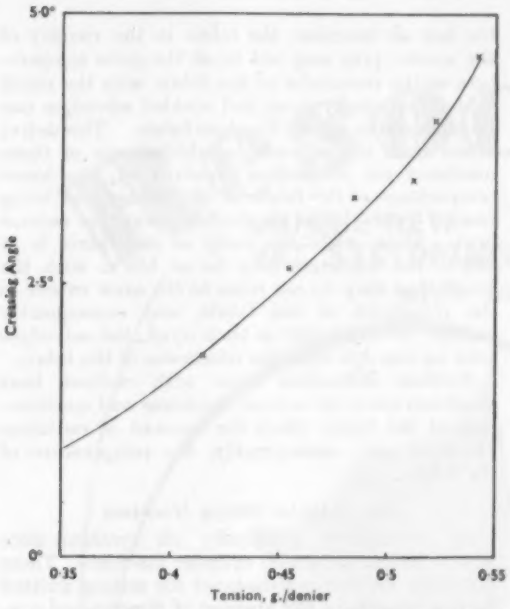

FIG. 7— Relation between Stiffness of Fabric and Tension Developed During Setting at Fixed Length

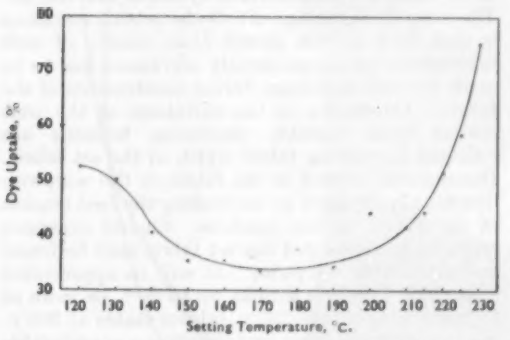

FIG. 8— Effect of Setting Temperature on Dye Uptake (2% Dispersol Fast Scarlet B 150 at 100°C. for 90 min.)

220°C. and above, is lower than that of the unset scoured fabric Fig. 8 shows the effect of setting temperature on the dye uptake of Terylene on dyeing at 100°C. for 90 min. with 2% Dispersol Fast Scarlet B 150, based on the fabric weight. Similar but less marked effects occur with other dyes. The increase in dye uptake above that of the unset scoured fabric at setting temperatures at 220°C. and above is thought to be associated with disorientation of the fibre; however, this is not reflected in the X-ray results obtained on unset and set fabrics (Table I).

TABLE I

Sample	Thickness*	Crystallinity†	Crystallite Orientation† $\theta°$	Overall Orientation* Δ
Scoured	69·0	Poor	6·2	180
Scoured and set at 120°C.	62·4	Poor	7·5	201
Scoured and set at 160°C.	59·9	Fair	7·5	213
Scoured and set at 220°C.	57·6	Good	7·5	235

* Data obtained from optical measurements
† Data obtained from X-ray measurements
θ = Mean angle of deviation from fibre axis
Δ = Birefringence

When taken together, these results do not present a clear picture of what is occurring when a fabric is set at different temperatures. It appears that the higher the setting temperature, the greater the overall orientation, yet the best crystallite orientation occurs in the scoured unset sample.

(e) *Other Properties*

Terylene fabrics may be set at temperatures up to 230°C. without substantially changing their physical properties or affecting the colour of the fibre.

2. THE USE OF AQUEOUS HEAT

(a) *Low-pressure Steam or Boiling Water*

Terylene filament fabrics, unlike those made from nylon, cannot be stabilised to elevated temperatures by low-pressure steam or boiling water treatments. From the known differences in the shrinkage behaviour of the two fibres Terylene would not be expected to react to these treatments in a manner similar to nylon. Thus, measurements of the boiling-water shrinkages of loom-state Terylene and nylon fabrics of a similar construction gave mean values for warp and weft shrinkages of 7% for both fabrics. However, whereas this relaxed nylon fabric was almost stable on subsequently exposing it to air at 200°C. and only shrank a further 3–4%, the Terylene fabric shrank a further 11–12% as the result of a similar treatment. The reason for this is clear from Fig. 1, in which it will be observed that unlike Terylene the shrinkage of nylon in boiling water and air at 200°C. are similar.

(b) *High-pressure Steam*

Although Terylene fabrics may be effectively stabilised by high-pressure torpedo steaming, this method of setting is not recommended since it causes a degradation of the fibre, by hydrolysis, which manifests itself in loss of tensile strength and discoloration of the fibre. The degree of degradation, as measured by the fall in tensile strength and change in intrinsic viscosity of the fibre, is dependent on steam pressure (Table II).

TABLE II

Steaming Time (min.)	Steam Pressure (lb./sq. in.)	Temp. (°C.)	Loss in Tensile Strength (%) Warp	Weft	Intrinsic Viscosity
0	—		—	—	0·61
20	40	141	Nil	Nil	0·57
20	75	160	Nil	12	0·51
20	100	170	4	18	0·46

It will be noted that intrinsic viscosity measurements, which are more sensitive than tensile measurements in recording fibre degradation, indicate that some degradation occurs even on steaming at 40 lb./sq. in. for 20 min.

A further disadvantage of torpedo setting is the inadequacy of control over fabric dimensions which

results in a set fabric, the width of which frequently varies from the outside to the inside of the batched fabric. Variations in physical and dye properties also occur due to temperature gradients through the fabric.

SETTING MACHINES

The two main types of setting equipment available in this country, which operate either on a batch or continuous principle, were developed for nylon fabrics, and their suitability for setting Terylene fabrics will now be considered.

Batch setting processes are represented by torpedo steaming methods, and as these have already been shown to be inadequate for Terylene fabrics they will not be considered further.

Heat-setting machines which operate on a continuous basis may be divided into two main groups —(*a*) pin stenter machines and (*b*) cylinder machines.

(*a*) *Pin Stenter Setting Machines*

On this type of machine, which may be used for both knitted and woven fabrics, the fabric is maintained under complete dimensional control during its passage through the hot zone. A noteworthy feature of this machine is an automatic fabric overfeed mechanism which presents the fabric to the stenter pins and also permits the fabric to be stretched, held at constant length or relaxed by fixed amounts as it passes through the setting unit. Pin stenter setting machines available, at present, for setting Terylene fabrics may be divided into two main classes— (i) those which use hot air and/or gases as the heating medium and (ii) those which use radiant heat.

(i) HOT AIR AND/OR GASES— Several firms market machines of this type, and although they differ in design details, their basic operating principles are similar. The pin stenter setting machine is similar to the conventional pin stenter, but whereas the latter normally operates at temperatures up to 150°C. the setting machines will operate up to 250°C. and above, if required. Terylene fabrics have been set at temperatures up to 230°C. on this type of machine and dyed to a good standard of quality. The even dye results obtained undoubtedly arise from the good uniformity of heating provided by the hot air blast which impinges on both sides of the fabric and rapidly equilibrates to the same temperature, both stenter pins and fabric as they pass through the hot zone. The temperature of the fabric in the hot zone is known with good accuracy.

(ii) RADIANT HEAT— Radiant heat is normally used in conjunction with a pin stenter and is an alternative, and possibly more economical, method to hot air, for raising the fabric to the desired setting temperature. A typical radiant heat setting machine consists of a pin stenter with a bank of infrared heaters set above, and corresponding reflectors located below the fabric path. The temperature of the heaters is thermostatically controlled but the temperature of the fabric in the hot zone is not known with any degree of accuracy. As in the hot air pin stenter machine, the fabric dimensions are accurately controlled, but unlike the hot air machine, the fabric in the vicinity of the stenter pins may not be at the same temperature as the remainder of the fabric with the result that differentially dyed and cockled selvedges can be produced in the set Terylene fabric. This defect arises from the manner in which some of these machines are at present constructed; the lower temperature of the fabric at the stenter pins being caused by the latter conducting away the radiant heat. Thus, when the body of the fabric is at 200°C. the selvedges may be at 150°C. with the result that they do not relax to the same extent as the remainder of the fabric, and consequently cockle. If the fabric is then dyed, the selvedges take up less dye than the remainder of the fabric.

Further difficulties arise with radiant heat machines since the colour, thickness and construction of the fabric affect the amount of radiation absorbed and, consequently, the temperature of the fabric.

(*b*) *Cylinder Setting Machines*

In the U.S.A. practically all synthetic-fibre woven fabrics are set on cylinder machines. These machines are entirely unsuited for setting knitted fabrics because of the absence of dimensional control. Fabrics are set by contact with hot cylinders or by using a combination of cylinders and hot air. The main disadvantage of cylinder setting machines is that they do not permit close control of weft dimensions and consequently allowance has to be made for weft shrinkage during construction of the fabric. Differences in the shrinkage of the weft arising from variable processing tensions are reflected in varying fabric width in the set fabric. Dimensional control of the fabric in the warpwise direction is obtained by controlling the feed tension of the fabric to the machine. Should excessive tensions be developed the set fabric may be found to be unstable warpwise. It will be appreciated that since Terylene shrinks up to 20% or more at temperatures of 200–230°C. fabrics stable at 200°C. may only be obtained by tolerating a considerable weft shrinkage.

HEAT SETTING CONDITIONS FOR TERYLENE FILAMENT FABRICS

Setting conditions on both pin stenter and cylinder setting machines are determined by the length of the hot zone or surface, which is substantially fixed by the machine design, on the moisture content of the fabric to be set and its thickness. It is not, therefore, possible to prescribe standard conditions for setting Terylene fabrics, but the data presented in Fig. 2 and 3 form a useful guide to establishing the optimum conditions and achieving the desired dimensional stability.

Terylene fabrics may be set at one of three stages— before scouring, after scouring but before dyeing, or after scouring and dyeing. There are obvious advantages in setting at the earliest stage in processing since subsequent wet operations will help to "break down" and restore the handle of the set fabric. Furthermore, wet creasing and shrinkage will be minimised and the curling of knitted fabrics reduced. The feasibility of setting before scouring

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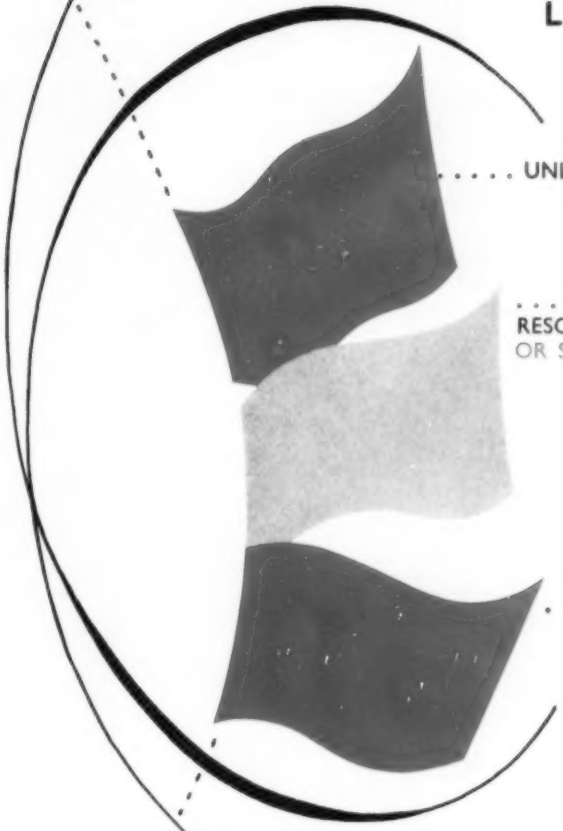

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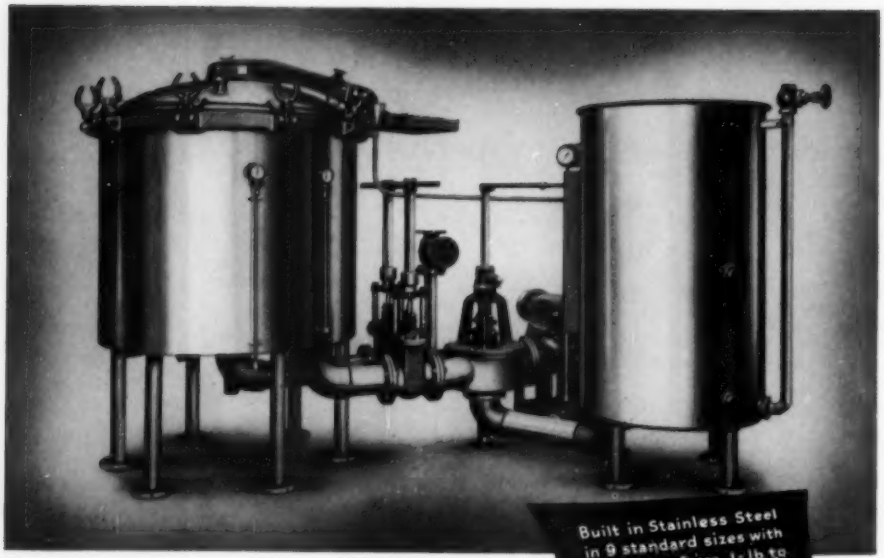

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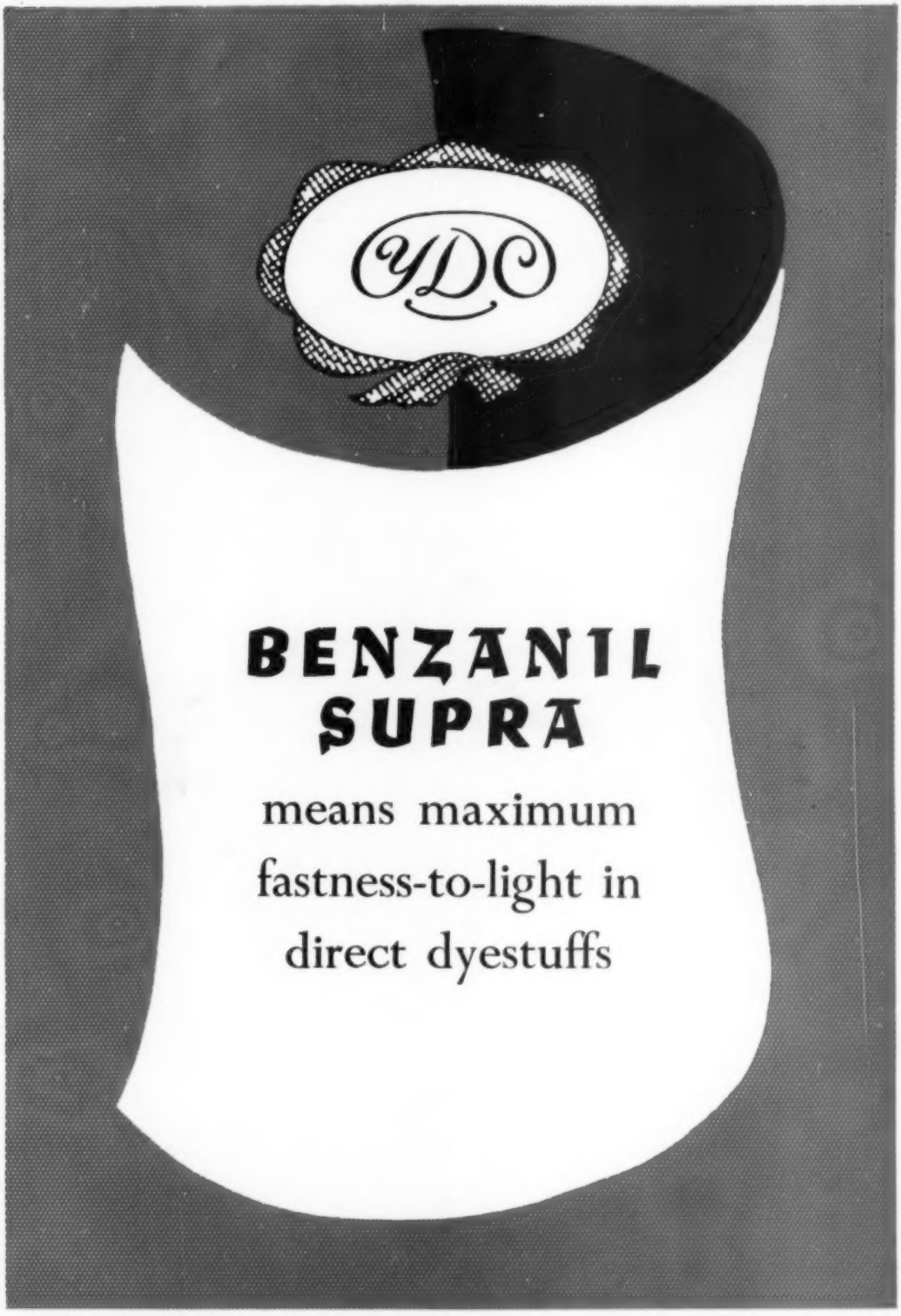

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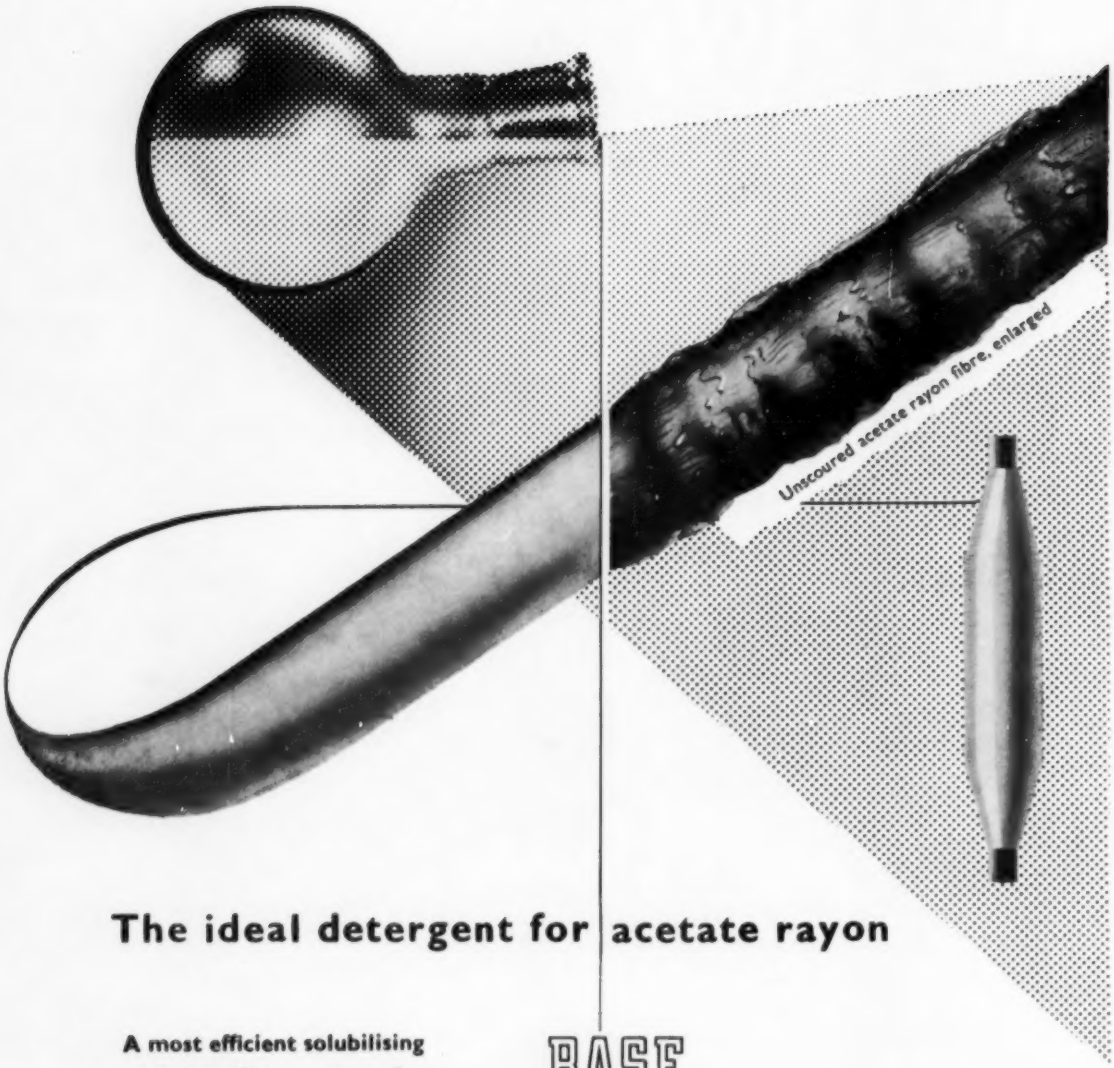

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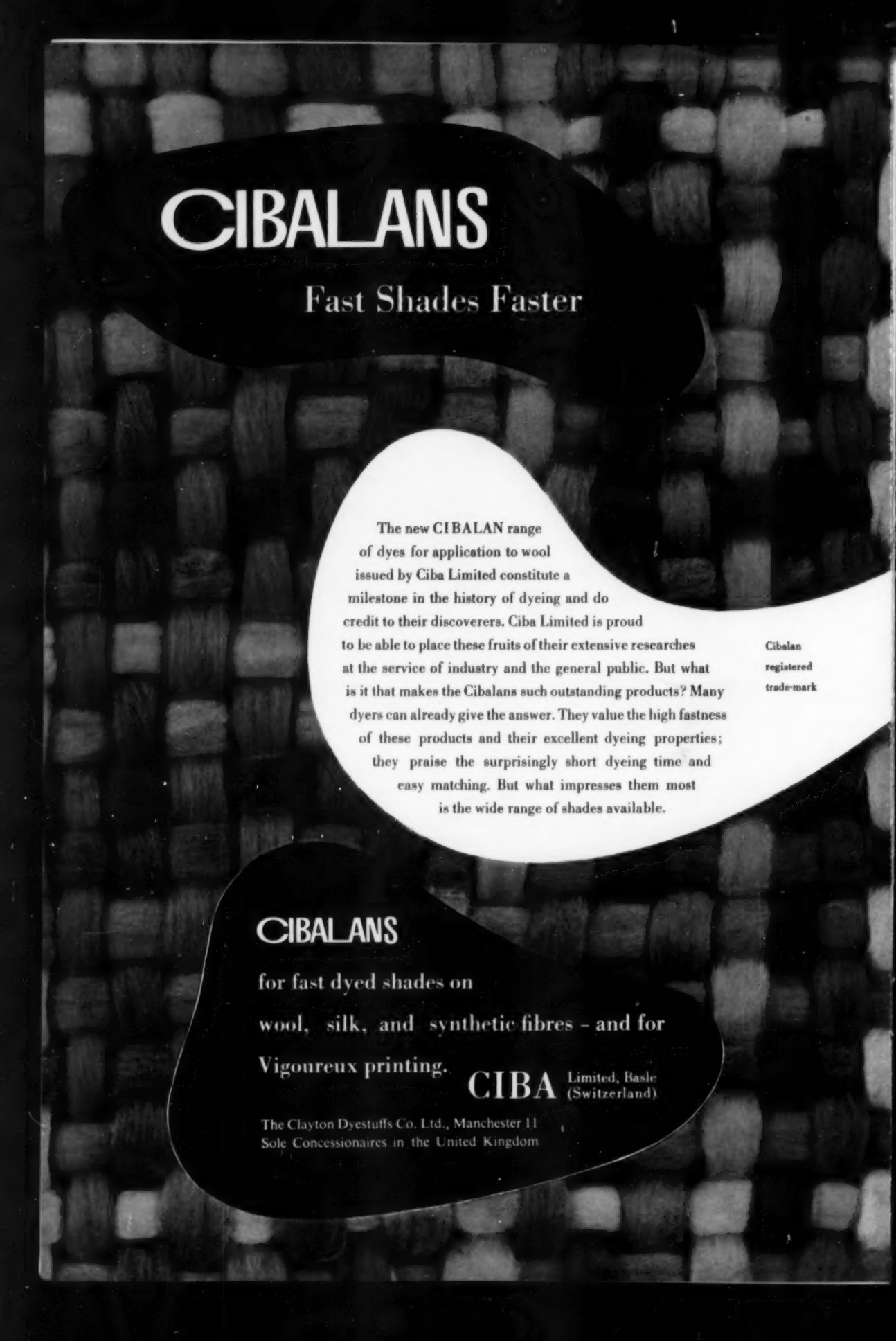
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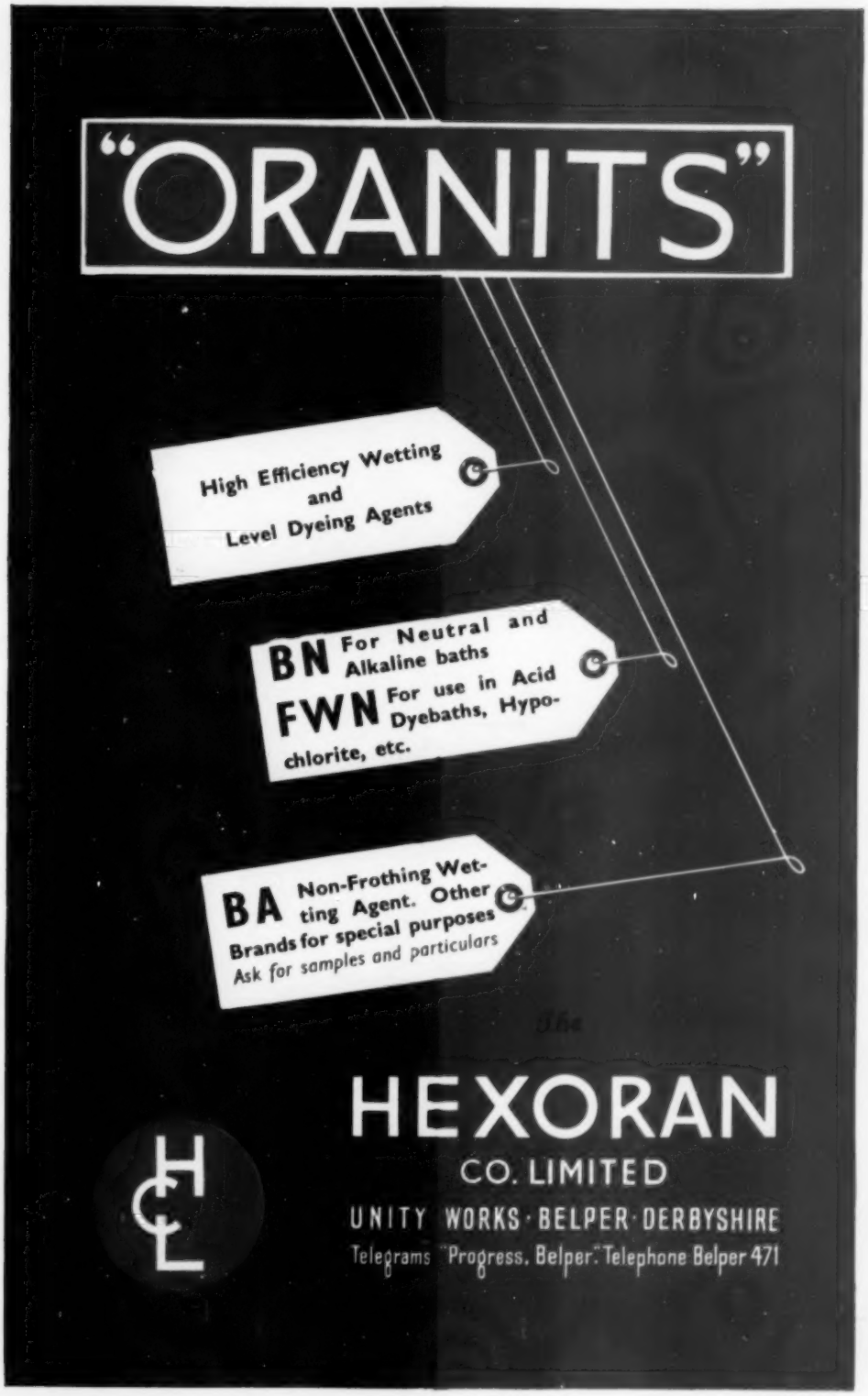
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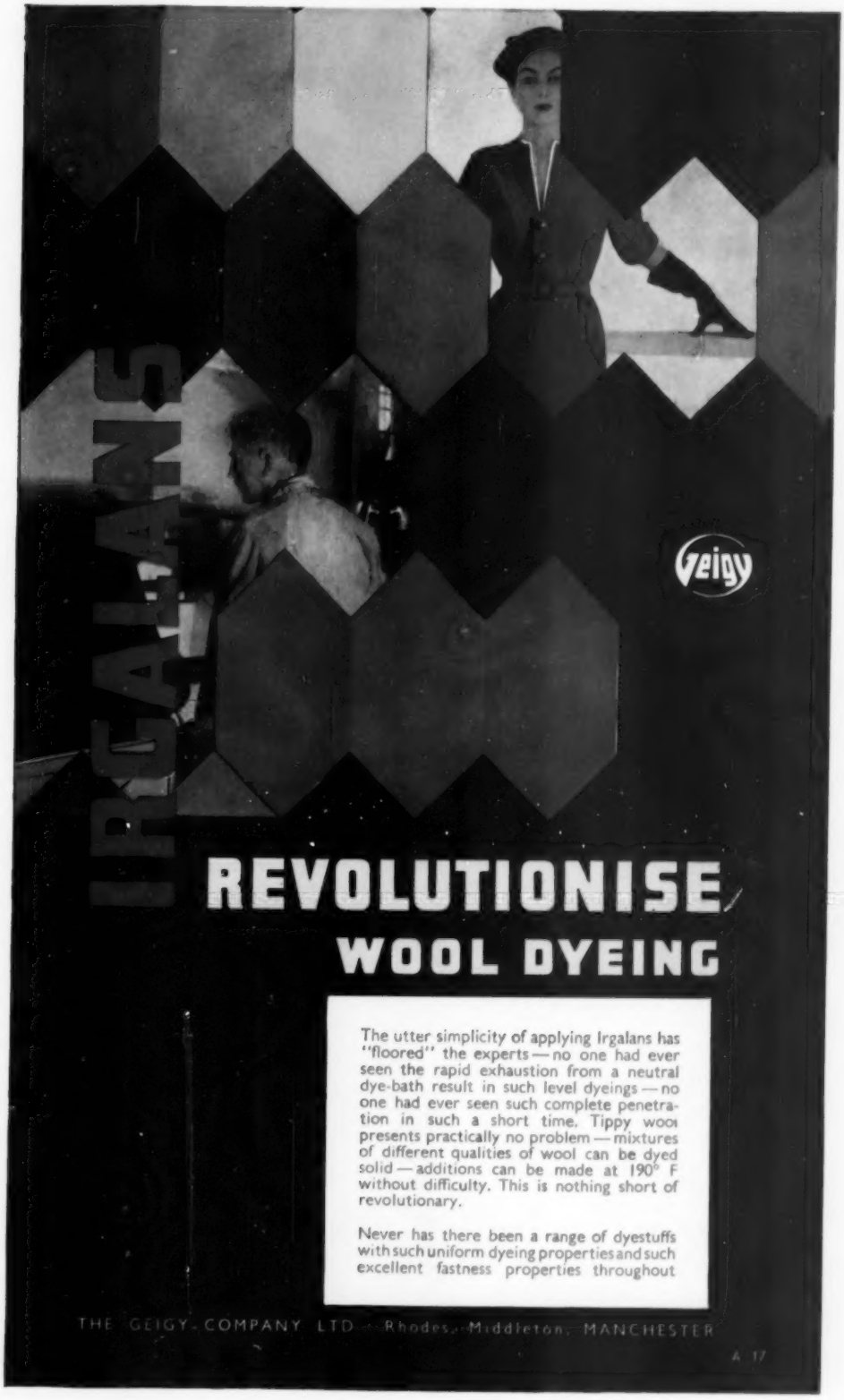
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Fabrics which are not set prior to scouring will shrink when scoured by an amount proportional to the temperature of the liquor and unless scoured in open width may also acquire creases which are difficult to remove. In these cases low-temperature scouring is advisable.

CONCLUSIONS

In this survey the effects of heat setting processes on the properties of Terylene filament fabrics and on subsequent processes, such as dyeing, have been displayed. Terylene polyester yarn is a new fibre with new properties and much work remains to be done, particularly in the field of finishing, before we obtain optimum properties in Terylene fabrics. Sufficient has, however, already been done to show that we can look forward to the unique properties of the fibre in the skilful hands of the finisher providing us with a new and exciting range of fabrics.

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(*MS. received 2nd April 1953*)

Discussion

Mr. J. T. MARSH: Why is it not possible for the yarn manufacturer to supply Terylene in the set state instead of leaving the setting to the finisher of the fabric?

Mr. MARVIN: Economics is the answer. However, would finishers like to have to finish Terylene fabrics which are dimensionally stable in the loom-state? We think not, and believe that the shrinkage characteristics of Terylene yarn as manufactured permit him to practise his skills to obtain a variety of effects in Terylene fabrics which he could not achieve if we supplied the yarn in a set, i.e. dimensionally stable, state.

Mr. C. P. ATKINSON: Would Mr. Marvin agree that, when Terylene fabrics are set on the pin stenter by radiant heat, there may be irregular presetting and variable affinity for dyes, because it is generally necessary to place the reflectors underneath the cloth? Reflectors are satisfactory only when they are kept absolutely clean and free from films of dust, etc.; otherwise their efficiency is appreciably reduced.

Mr. MARVIN: The defects in the form of unevenly dyed and cockled selvedges which have occurred in Terylene fabrics set on radiant heat setting machines have been caused in the main by the stenter pins, which conduct away the heat from the fabric selvedges, which are, therefore, at a lower temperature than the remainder of the fabric. It may of course be possible to overcome this defect by modification of machine design. It is true that reflectors which are used in conjunction with radiant heaters lose their efficiency unless they are kept clean.

Mr. J. H. LEACH: What is the effect of dry heat setting on the relative efficiencies of carriers during dyeing with dispersed acetate-rayon dyes?

Does the effect of heat setting negative in any way the dye uptake improvement brought about by such carriers as 2-hydroxydiphenyl?

Mr. MARVIN: The dye uptake of heat-set Terylene fabrics is increased by employing carriers such as 2-hydroxydiphenyl when dyeing with disperse dyes. The effect of setting temperature on dye uptake, recorded for dyeings in the absence of a carrier and demonstrated in my paper, also obtains when dyeing with a carrier. The dye uptake is, however, correspondingly higher with a carrier.

Mr. E. BOTTOMLEY: Can the lecturer indicate whether creasing encountered during scouring can be eliminated during subsequent hot-air setting on a pin stenter?

Mr. MARVIN: The creasing of Terylene fabrics during scouring should be avoided by scouring at low temperatures. Where creasing has occurred, however, no difficulty has so far been encountered in removing the creases during setting on a hot-air pin stenter. However, it will be appreciated that, if creasing is also accompanied by thread displacement, the latter may not necessarily be corrected by setting the fabric on a hot-air pin stenter.

Dyeing of Nylon Yarn in Package Form

W. S. WILLSON

One-day Symposium on Synthetic Fibres held by the Manchester Section at the Manchester College of Technology on 13th March 1953, Mr. F. Farrington in the chair

A method of dyeing continuous-filament nylon yarn in cake form has been developed and proved on a bulk scale. The "mock" cakes are wound on a converted coning machine, and dyeing is carried out on standard package-dyeing machinery. The use of various classes of dye is discussed, and suitable technique and aftertreatment are described.

1. INTRODUCTION

The work which is reported here on the dyeing of nylon continuous-filament yarn in package form was started three years ago in order to overcome the difficulties which were met in handling such yarn in hanks. The fine deniers in which nylon is produced, and the low specific gravity of the fibre, made it virtually impossible to treat it in hanks on the conventional rotating-arm machines of the Gerber type. Floating, tangling, and waste in backwinding made the process tedious and uneconomic.

In considering package dyeing, two possible methods were examined—(*a*) cheese or cone, (*b*) cake. Nylon filament yarn is produced on a bottle bobbin which contains 1 lb. of yarn. This bobbin is very hard and contains yarn which is still under some strain, so that even if the package holder were perforated and of suitable dimensions for a dyeing machine, it would be quite impossible to penetrate the yarn. Moreover, there is a potential shrinkage of 8% in length when this yarn is immersed in boiling water. The first trial made was, therefore, directed to producing a cone on a perforated Longclose centre, of sufficient softness to allow penetration.

A Leesona No. 50 coning machine was used, adapted for pineapple coning and using a $2\frac{3}{4}$ honeycomb wind, and an 8-oz. cone was produced of low-twist yarn ($\frac{3}{4}$ t.p.in.). The first attempt to dye this was a failure because of shrinkage, tightening of the yarn, and lack of penetration. In order to overcome shrinkage during dyeing, the yarn was pre-shrunk by steaming under 25 lb./sq. in. pressure and then rewound on to a cone as before. The result in this case was fairly satisfactory for levelness and penetration, but the process was long and expensive. It had, however, established the necessary conditions and the situation could be examined.

Most nylon yarn for knitting or weaving is required in thrown condition, 7-30 t.p.in. being used according to the end use. Such thrown yarn is twist-set by a mild steaming process, which subdues liveliness and also reduces potential shrinkage to 1–2%. It was found that such yarn could also be coned or cheesed and dyed on perforated centres, but the results were not completely satisfactory for levelness. The use of Franklin-type springs was found to give improved results, since any shrinkage can take place without tightening unduly. Moreover, it was not certain that the steaming process previously referred to, of pre-shrinking in cheese form, was giving complete and even shrinkage and dyeing troubles might arise from this irregularity. As an alternative method, and in view of the well established process of cake dyeing on viscose, it was decided to examine possibilities of making a mock cake, i.e. a centreless package similar in form and dimension to the viscose cakes produced during spinning. It was thought that by using such a package of yarn which had been twist set only, the small shrinkage would be accommodated without undue tightening either within the mock cake or on the dyeing former.

2. CAKE WINDING

It was found quite easy to make such a mock cake, and the process offered the following advantages—

(i) 1 lb. of yarn can be put on a package against 8–12 oz. on cheese.

(ii) It is quicker to wind a cake using the same spindle speed, since the starting diameter is 4 in. against 2 in. for cheese.

(iii) The large-diameter package is more free to accommodate itself in regard to shrinkage or spreading on the machine.

(iv) The package is softer and more easily penetrated—the wall thickness of yarn is about $2\frac{1}{4}$ in.

It may, indeed, be possible to make a larger cake than the 1 lb. which has been standardised throughout this work. This has been done with coarse yarns by doubling the traverse length during cake winding and so making a package 6 in. long with 4 in. inside diameter. Such a variation could probably be applied to finer-denier yarns if it offered any economic advantage.

Since the manufacture of the cake is perhaps the most important operation in the process, this is described in detail.

A No. 50 Leesona cheesing machine was fitted to make a cake with 4 in. internal diameter and 3 in. traverse. This fits the $3\frac{1}{2}$ in. × $3\frac{1}{2}$ in. individual Bakelite or stainless steel former on the Longclose or Courtauld machine, allowing the cake to spread endwise, and to shrink radially if necessary. The normal cheese mandrel was replaced by a mandrel made from 16-gauge steel, 4 in. outside diameter,

and capable of being expanded or contracted to facilitate doffing. A roller bail was fitted in place of the button guide, and all tension devices were removed from the machine. Suitable gears for a 2¾-honeycomb wind were fitted and the 4 in. mandrel was covered with a cotton stockinette.

The thrown yarn on uptwist cheese is wound direct from package to traverse guide, using only a pigtail take-off above the supply package. In this way, at a spindle speed of 1,200 r.p.m., a 1-lb. cake can be wound in 5 hr. with 30-denier yarn. The weight of the cake is regulated by doff times and it is preferable to use 1-lb. packages all through, where this is possible. The outside diameter is about 6¼ in. and the package is uniformly soft and must be handled carefully.

When the mock cake has been wound, the stockinette is drawn round the ends and face of the package and secured firmly by twisting the loose portion of stockinette and tucking it into the overlap. The mandrel is then collapsed and the mock cake removed carefully and stood on its end. To preserve its circular shape, a coiled sheet of thin resilient bakelite is placed in the hollow centre of the cake and allowed to expand radially, holding the cake firmly. The cakes are packed in separate compartments in cartons for transport to make sure that no distortion takes place during conveyance. A similar procedure is adopted for handling the cakes after dyeing when returning them for back-winding.

3. DYEING

The manufacture of the mock cake has been described at some length, since it constitutes a very important part of the process. The redistribution properties of most classes of dye are poor on nylon and it is essential to start with a package which allows quick and even liquor flow for good results.

Dyeing can be carried out either on the Courtauld machine, the cakes being packed end to end on fluted tubes or individual formers held horizontally, or on the Longclose machine, in which case the cakes are placed on a 3¼ in. × 3½ in. Bakelite or steel formers which are stacked vertically on the spindle.

The yarn on package is first scoured with the usual detergents— soap or synthetic— plus alkali, to remove any finish or size, and is then rinsed. Dyeing can be done with any of the usual classes of dye used for nylon and the application of individual classes will be dealt with later. At present the best sequence of operations is discussed.

It has been found that there is no advantage to be gained by reversing the flow of liquor, and indeed by maintaining it from inside to outside, the cake is kept in better condition. After dyeing, it is preferable to use a single spindle hydroextractor, on which a complete spindle of 6–8 cakes can be hydroextracted without removing the cakes. If necessary, the cakes could be removed, flattened, and hydroextracted in a basket centrifuge, but extra handling is always undesirable. After hydroextracting, the cakes are removed, bakelite inserts replaced and the cakes are dried in the stove. Drying is much quicker than with viscose or cotton and can be achieved overnight at 70°C.

Whatever class of dye is used, it has been found best to finish off after normal rinsing with a final bath containing 2 g. Lubrol W (ICI) or other antistatic agent and 1 g. gelatin per litre at 40°C. The anti-static agent is of great assistance during back-winding in preventing ballooning due to static, and in keeping the yarn under control. The gelatin imparts a slight firmness to the cake, and by lightly binding the threads together, prevents sloughing and distortion during transport, handling and back-winding. These additions are easily removed during scouring of the knitted or woven fabric.

Back-winding of the cake on to pirn or cone can be accomplished on standard machines winding overend from the cake by the usual viscose rayon technique. At 1200 r.p.m. coning can be carried out with less than 1% of waste for all deniers. It can be assumed from this that the process mechanically is satisfactory, and it has been in use now on bulk scale as an economic commercial proposition.

4. FASTNESS AND TYPES OF DYE

Since most dyed yarns will be used for stripes, checks, and effect threads in coloured woven or knitted nylon fabrics, it is usually necessary to achieve sufficient fastness to stand up to subsequent setting processes, which may include boiling water, dry heat at ca. 420°F., or even pressure steam. Most yarn is, therefore, dyed with acid or chrome dyes, according to end use. Acid dyes of the Carbolan (ICI) type have good all-round fastness to severe scouring and even boiling, and can be applied by the normal technique. Control of dyeing is by means of temperature or pH according to the requirements of the dye, and it is not possible to lay down any universal rule. Each dye has its peculiarities, and success depends on applying it slowly and gradually. Exhaustion is good, up to the limit of saturation of nylon by the acid dye.

For better fastness, the chrome or chrome-complex (Neolan or Ultralan type) dyes may be used. The Neolan (Ciba) or Ultralan (ICI) dyes are usually applied to wool from a bath containing 8% sulphuric acid, and such treatment at the boil may result in some degradation of nylon yarns. Satisfactory results are obtained on nylon by using 1–5% formic acid, or even acetic acid, in place of sulphuric acid. It should also be mentioned that some of these dyes are sensitive to alkaline scour, and should be finished off with a boiling soap to allow for this.

A range of dyes can be selected which will stand up to pressure steaming at 25 lb. sq. in. for 2 min. and such dyes are used for coloured heels on stockings which have to be preboarded. Slight bleeding may take place if the steam is very wet, but when the stockings are overdyed to mode shades, this is not serious. It can be cleared, if necessary, by a mild detergent and Formosul treatment.

The new ranges[1] of metal-complex dyes which are now available have been tried out and provide

useful dyes of good light fastness which are not difficult to apply in a neutral bath. Dyeing can be slowed at the start by the addition of ammonia.

Vat dyes have been dyed by this means with good results, but in view of the unsatisfactory fastness and colour value which are frequently found with this class of dye, very little work has been done. The method used was the normal reduction technique, using dextrin or glucose where necessary to prevent breakdown of the leuco compound at the high temperature necessary in applying vats to nylon[2]. The Abbot–Cox method[3] has not been tried yet. It is known that re-oxidation of leuco vat dyes on nylon is slow, and in package form an oxidising rinse must be used, perborate or dichromate being suitable. As mentioned before, the vat dyes are expensive, and many have poor light fastness, so that their use would be limited to functions where excellent wet fastness alone was of supreme importance. Ingrain socks which have to be preboarded provide a possible outlet.

For similar reasons, the azoic dyes have not been investigated and a good bright scarlet of suitable fastness is awaited.

One point deserving mention is the inability of anionic dyes to cover inherent dyeing differences in nylon yarn. This defect, while still present, is much less prominent than in the early days of nylon production. By using only first-grade weaving yarn, which has been merged for good dyeing properties, it is possible to produce results which are of reasonable evenness and quite suitable for narrow stripes or checks, effect threads, stocking heels, small-pattern jacquard fabrics, etc. At this stage it would be undesirable to put dyed nylon yarns into solid woven fabrics. Indeed, it is risky with any continuous-filament yarn, which is far more critical than spun yarn because of the sheer nature of the fabric produced.

5. STRIPPING

It sometimes happens that a batch has to be stripped for re-dyeing. With nylon this is admittedly difficult in view of the generally good wet fastness of acid dyes on nylon. Disperse dyes can readily be stripped from cakes by means of Formosul, but acids and chromes require a stronger treatment. Sodium chlorite has been used successfully on cakes, but its effect on stainless-steel machines is serious. The Solvay process[4] using aluminium strip or cobalt oxide is a palliative, but a simpler method is still sought.

6. STAPLE YARN

Spun nylon yarns can be dyed successfully in hank on the Gerber-type machine, in the same way as cotton, though some trouble may be experienced with floating in the dyebath. To overcome this, package dyeing is very effective and can be done either in cheese or mock cake form. No modifications are necessary in cheese dyeing and use of the Franklin type spring centre has given excellent results. Here again, the cake can be used with equal success, but since this represents a departure from normal spun yarn procedure, it is doubtful whether it will gain much popularity. The suitability of dyed cheeses or cones for knitting or warping without back winding may be a factor in their favour. The field for spun nylon yarns is not limited by considerations of inherent dyeing differences in the basic fibre, since these fibres are blended in spinning, and given uniform staple to start with, the results are uniform. The acid and chrome dyes will fulfil most requirements in this trade.

In the field of vessel dyeing, though not strictly concerned with packages, it should be mentioned that loose nylon staple can be dyed satisfactorily in the Obermaier machine, and tops and slubbing are regularly dyed in the usual type of plant employed for wool. No modifications are necessary, beyond observing the careful control of dye application, and replacing the anti-static dressing on the fibre prior to drying for combing or spinning.

* * *

Acknowledgment is made with thanks to Messrs. British Nylon Spinners Ltd. for permission to publish this paper and also to Messrs. Courtaulds Ltd. for their assistance at Droylsden and Coventry in carrying out trials and bulk dyeings.

(*MS. received 14th April 1953*)

References

[1] Cibalan (Ciba), Vialon (Basf), Capracyl (DuP), Irgalan (Gy), etc.
[2] *Amer. Dyestuff Rep.*, **38**, 213 (1949).
[3] *BP* 593,008.
[4] *BP* 640,394.

Discussion

Mr. A. H. NORRIS: What is the time of steaming at 25 lb./sq. in. for unset nylon?

Mr. WILLSON: In coning yarn for dyeing an arbitrary time of 30 min. was used in the first instance to bring about shrinkage, but, as mentioned, we were not satisfied with the process. It is not now used on account of the possibility of unevenness.

Mr. NORRIS: What, if any, lubrication is carried out?

Mr. WILLSON: This is a matter for the winder, and depends on the end use of the yarn. For knitting a vegetable–mineral oil may be used, but for warping or pirning naturally no lubrication is used. The dyer would use only the finish mentioned in the text.

ERRATUM

Lanasyn Yellow 2RL (S) (J.S.D.C., **69**, 452 (Nov. 1953))— Owing to a misprint this was erroneously spelt "Tanosyn".

Notes

Proceedings of the Council

At meetings of the Council held at the offices of the Society, 19 Piccadilly, Bradford 1, on 21st October, 18th November, and 9th December 1953, the proceedings included the following items of interest—

SYMPOSIUM ON TEXTILE PRINTING— A report on the Symposium was adopted with congratulations and best thanks to the Symposium Committee and others associated with its organisation.

DECEMBER JOURNAL— Approval was given to the suggestion of the Publications Committee that an ordinary issue of the *Journal* be published in December as well as the Proceedings of the Symposium on Textile Printing.

MEMBERSHIP— Fifty-eight applications for ordinary membership and fourteen for junior membership were approved.

DYERS COMPANY MEDAL— The report of the Adjudication Committee of the Worshipful Company of Dyers Research Medal, recommending the award of the Research Medal for 1952–53 to Dr. R. H. Peters, was accepted (see p. 6).

SOCIETY'S MEDALS— The recommendations of the Society's Medals Committee for the award of medals for 1953 were accepted (see p. 4).

SOCIETY'S MEDALS COMMITTEE— It was resolved that the Convener of the Worshipful Company of Dyers Medal Adjudication Committee be *ex officio* a member of the Society's Medals Committee.

PERKIN CENTENARY COMMITTEE— It was reported that Dr. C. J. T. Cronshaw and Mr. R. J. Smith had accepted invitations to serve on this Committee, and Dr. Cronshaw had agreed to act as Chairman.

INTERNATIONAL FEDERATION— After full discussion of the question of membership of the International Federation of Associations of Textile Chemists and Colourists, it was resolved that the matter be allowed to lie on the table.

BADGE FOR RETIRING PRESIDENTS— It was resolved that arrangements be made to present a badge to retiring Presidents.

FELTMAKERS RESEARCH MEDAL— It was reported that, as no papers on feltmaking had appeared in the issues of the *Journal* from July 1952 to June 1953, again no award of the Worshipful Company of Feltmakers Research Medal could be made.

VICE-PRESIDENTS— A tentative scheme for the changeover from twelve Vice-presidents serving for three years to six Vice-presidents serving for six years each was accepted for submission to the Annual General Meeting in 1954.

NOMINATION OF PRESIDENT— Mr. F. L. Goodall was nominated President for 1954–1955.

NOMINATION OF HONORARY TREASURER— Mr. H. Jennison was nominated Honorary Treasurer for 1954–1955.

NOMINATION OF HONORARY SECRETARY— Mr. J. G. Hopkinson was nominated Honorary Secretary for 1954–1955.

CHEMICAL SOCIETY JOINT LIBRARY COMMITTEE— Dr. T. H. Morton was nominated to represent the Society on this Committee during 1954.

Meetings of Council and Committees December 1953

Council— 9th
Publications— 15th
Colour Index Editorial Panel— 16th
Terms and Definitions— 4th
Society's Medals— 3rd
Joint Conference Committee— 11th

Deaths

We regret to report the loss by death of Mr. J. Faulkner, Mr. D. Morton, Mr. R. Ritchie, and Mr. Arthur E. Roberts.

A.S.D.C. Examination

It is intended to hold an examination for the Associateship of the Society of Dyers and Colourists (cf. J.S.D.C., **69**, 394–395 (Nov. 1953)) toward the end of 1954. Further details will be announced later. Meanwhile, those who wish to take the examination in 1954 are requested to register their names with the Society **before 31st March 1954,** so that the necessary arrangements can be made.

Midlands Section Exhibition

An Exhibition of "Scientific Aids to the Dyeing Industry" will be held in the Great Hall, Leicester College of Technology, on Friday afternoon and Saturday, 28–29th May 1954. The purpose of this will be to show and to demonstrate, both to students and to dyers generally, a wide range of instruments available for the control of quality and production. A large number of manufacturers of such instruments have already promised apparatus and demonstrators.

Fuller particulars will be published later. In the meantime, any member or firm who would like to offer an exhibit is invited to communicate with the Honorary Secretary of the Exhibition Committee, A. Datyner, Esq., The College of Technology, Leicester.

Trelon Polyamide Fibre

Trelon (Thüringisches Kunstfaserwerk, Schwarza, Eastern Zone of Germany) is a new polyamide fibre, for which lignite or furfural is the raw material. It has m.p. 20°C. higher than that of Perlon and is highly resistant to cold and hot caustic alkalis and most dilute acids but is destroyed by free chlorine. It is soluble in formic acid, concentrated sulphuric acid, phenols, and hot concentrated acetic acid. It is said to be softer and smoother than other polyamide fibres and their equal in mechanical strength. C O C

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Fuel Conservation in the Textile Industry. *Dyer*, **110**, 397–400 (18 Sept. 1953).

Extract from the productivity report "Fuel Conservation", British Productivity Council. Comparison of British and U.S.A. textile finishing industries with regard to steam raising plant and fuel economy measures. U.S. practice favours the use of water tube boilers with automatic firing and it is stated that no difficulty is encountered in following the fluctuating steam load. In this country the Lancashire boiler is in general use owing to its ability to meet varying steam demands which are probably greater here than in the U.S. where continuous processing techniques are more common. In the U.S. efficient lagging of totally enclosed dyeing and drying machines is noted and also the widespread use of effluent heat exchangers. There is a description and drawing of a new hot air stenter by the National Drying Machinery Co. on which gas is used as the heating medium. It is considered that the most up to date works in Britain suffer little in comparison with U.S. plants but that as a younger industry the U.S. has a higher proportion of new works and new machinery. A. H.

Contact Electrification—Production and Dissipation of Static. P. S. H. Henry. *Science Progress*, (164), 617–634 (Oct. 1953).

A review of the literature (50 references) is followed by a brief account of the troubles it can cause and methods of avoiding them. C. O. C.

PATENTS

Thread Processing Reels. Courtaulds. *BP* 698,932

The reel has a hollow shaft through which a heating medium is circulated. This shaft has carbon-graphite bearings and part of the heating medium is directed to act as a lubricant for these bearings. C. O. C.

Continuous Treatment of Thread, etc. J. M. Goppell. *BP* 699,675

The material is carried through a U-tube by the flow of liquor. The bent part of the tube is flexible and can be detached from one or both of the arms. When entering the material into the tube, the flexible portion of the tube is detached from the delivery arm and allowed to hang down straight so that the thread or other material is readily passed through it. Finally the flexible portion is reconnected to the delivery arm and the material carried up the delivery arm as the flow of liquor is resumed. C. O. C.

Wet Processing of Yarn Cakes. Celanese Corpn. of America. *BP* 698,784

Each cake is enclosed in several permeable coverings and is then wet processed and dried. The outer covering is preferably a textile fabric, e.g. a tubular knitted sock, which affords adequate protection during wet processing and drying. The inner covering is preferably of paper which suffices to protect the cake during handling, storage and transportation. C. O. C.

Wet Treatment of Warps. N. V. Onderzoekingsinstituut Research. *BP* 698,728

The warp is passed in open width through a trough the threads of the warp being so spaced that the outermost are $\pm$ 1 cm. from the side walls of the trough. The trough has a false bottom. The liquor is caused to flow regularly and not back into the part of the trough through which the warp moves by combined use of thread guiding rollers, either wholly or partly immersed, under which the warp passes, and curved baffles at the beginning and end of the trough which guide the liquor to or from the space beneath the false bottom. C. O. C.

Producing Vibrations in Liquids. A. Kärcher. *BP* 699,480

The vibrations are produced by the combustion of fuel and a one-way valve, the vibrations being caused to vibrate a diaphragm placed in the liquid. The device can be used to simultaneously heat and vibrate the liquid. C. O. C.

Multi-roller Felting Machine. M. Casse. *BP* 699,247

A multi-roller felting machine has two beds of rollers which impart to the material being felted reciprocating movements along an undulating path. Each roller is connected to a worm wheel and a worm is in mesh with the set of worm wheels belonging to each bed of rollers. There are two drives, one to rotate the worms and the other to impart an axial reciprocating movement to each worm, this reciprocatory movement causing the co-operating worm wheels and their respective rollers to oscillate about their axes and being superimposed on the rotary movement imparted by the first drive. C. O. C.

Felting. L. S. Adams Engineering. *BP* 699,090

The material while wet and hot is passed between rollers one at least of which is vibrated at a frequency of several thousands per minute. This speeds up hardening, milling and similar operations. C. O. C.

Automatic Measurement and/or Regulation of the Moisture Content of Textiles or like Materials. John Dalglish & Sons. *BP* 698,862

The moisture sensitive portion of a humidity detector is placed close to the material so that changes in humidity of the air at the surface of the material are immediately detected, the output of the humidity detector being used to effect automatic adjustment of the drying machine. C. O. C.

Coating Paper. Champion Paper & Fibre Co. *BP* 698,550

The paper web is supported, while the freshly applied coating material is smoothed out by a doctor blade, on a moving web-carrier of which at least the outer layer is formed of yielding resilient material. A film of water is continuously applied to the web carrier before application of the paper. A roll pressed against the surface of the web-carrier is adjustable to regulate the thickness of the water film. The doctor blade indents the surface of the carrier to elongate the surface in the immediate vicinity of the blade.

BP 698,610

The blade and web-carrier are wider than the web and the end of the blade beyond the web contacting portion slopes away from the surface of the carrier. The wiping surface of the blade is parallel to the web-carrying surface in a transverse direction and converges slightly towards it, at an adjustable angle, not exceeding 10°, in the direction of its movement. In addition to the wiping surface the blade has a surface forming a large angle with the web-carrying surface for removing excess coating material. S. V. S.

Treating Leather and other Pliant Sheet Material with Liquid. F. Lines and W. V. Cross. *BP* 699,688

The skins or sheets are carried in a horizontal plane through a treating chamber, where, e.g. they may be sprayed, and then through a drying chamber. Much more uniform coating of the leather is achieved than when the skins are treated while hanging down vertically. C. O. C.

Static Electricity Eliminators. Minister of Supply. *BP* 698,779

A container is filled with krypton 85 the container being such that the β-particles from the krypton reach the outer surface with an absorption in the body of $< 60\%$. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Cellulose Carboxydextrins in Soap Boiling. A. P. Antŷkov. *J. Appl. Chem. U.S.S.R.*, **24**, 848–853 (Aug. 1953).

In the preparation of soap part (10–30%) of the fat is replaced by carboxydextrins, which are prepared from lignin-free cellulosic material by air oxidation at the boil in presence of alkali followed by treatment with acid. The product is cheaper, and has improved detergent and foaming properties. A. E. S.

Interfacial Activities and Porphyrin Contents of Petroleum Extracts. H. N. Dunning, J. W. Moore and M. O. Denekas. *Ind. Eng. Chem.*, **45**, 1759–1765 (Aug. 1953).

A Californian crude oil was fractionated by solvent extraction and chromatography, and the interfacial activities, film forming tendencies, concentration of free porphyrin and metal-porphyrin complexes of the various extracts were determined. The metal porphyrin complexes were decomposed and then re-synthesised. It is confirmed that the interfacial activity of a crude oil is associated with its asphaltic content. A considerable amount of nickel-porphyrin complex and a smaller amount of vanadium-porphyrin complex was found in this crude oil and it is shown that these are largely responsible for the interfacial activity. The stability of the Ni and Va complexes accounts for the difficulty in removing these metals from crude petroleum. The presence of porphyrins is indicative of the biological origin of the petroleum. W. K. R.

Thermodynamics of Metal Chelate Formation. I. The Third and Fourth Dissociation Constants of Ethylenediaminetetraacetic Acid. F. F. Carini and A. E. Martell. *J. Amer. Chem. Soc.*, **75**, 4810–3 (5 Oct. 1953).

The potentials of the hydrogen–silver–silver chloride cell were measured in buffer solutions containing acid salts of ethylenediaminetetraacetic acid. The e.m.f. data obtained without liquid junction were extrapolated to infinite dilution using the Debye–Huckel limiting law and the thermodynamic equilibrium constants associated with the dissociation of the third and fourth protons determined. The standard free energy changes for these reactions at 0, 5, 10, 15, 20, 25 and 30°c. are given together with the corresponding values of ΔH° and ΔS°. C. O. C.

Stability of Metal Chelates. VII—*NN'*-Ethylenediaminedipropionic Acid and *NN'*-Ethylenediaminetetrapropionic Acid. R. C. Courtney, S. Chaberek and A. E. Martell. *J. Amer. Chem. Soc.*, **75**, 4814–8 (5 Oct. 1953).

The successive acid dissociation constants of *NN'*-ethylenediaminedipropionic acid and of *NN'*-ethylenediaminetetrapropionic acid and the stability constants of the 1:1 chelates of Cu (II), Ni (II), Co (II), Fe (II), Fe (III), Zn (II), Cd (II), Mn (II) and Mg (II) ions are compared with the corresponding stability constants of the analogous chelates of the ethylenediaminediacetate, -tetracetate and -diacetatedipropionate anions. For all metals investigated replacement of an acetate group by a propionate group decreases the affinity of the ligand for metal ions. The ferric chelates differed from those of the divalent metals in that mono- and dihydroxy derivatives were formed. C. O. C.

PATENTS

Detergent. Henkel & Cie. *BP* 699,571

A mixture of $< 60\%$ of soap and 2–10% of magnesium silicates colloidally soluble in water, the remainder being other detergent compounds but excluding solvents, oxygen-yielding substances and soda or substances more alkaline than soda, does not form precipitates with hard water.

BP 699,572

A mixture of soda and 2–10% of magnesium silicates colloidally soluble in water, is used. Neutral salts, oxygen-yielding substances, foaming agents and small amounts of salts of such phosphoric acids as contain less water than orthophosphoric acid may also be present. C. O. C.

Lubricant for Viscose Rayon. Courtaulds. *BP* 698,680

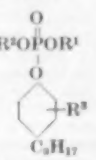
A lubricant for application to freshly-coagulated viscose rayon and which also prevents formation of rock-like deposits on thread guides, thread-advancing devices, etc. consists of solution in oil of a polyethyleneglycol ester of a long chain fatty acid of > 10 C or a polygylcol ether of such an acid. C. O. C.

Antistatic Agents for Use with Synthetic Resins. Chipcopee Manufacturing Co. *USP* 2,628,176

Long chain fatty acid partial esters of hexitol anhydrides and hydroxy-polyalkylene ethers thereof are antistatic agents which have excellent affinity for Saran, polyethylene and similar products. Some of them are readily dispersible in water but impart an antistatic finish which resists scrubbing and washing. C. O. C.

***p*-Tertiary Octylaryl Phosphates Emulsifying Agents.** ICI. *BP* 699,080

Compounds of formula—



(R^1 = Alk of < 4 C which may be substituted by halogen; R^2 = H, alkali metal, monovalent non-metallic base, or Alk of < 4 C; R^3 = H or Alk) are emulsifying agents for forming aqueous emulsions of mineral oils and other liquid hydrocarbons. C. O. C.

Trihydroxy Polyoxyalkylene Ethers of Glycerol. Dow Chemical Co. *BP* 699,664

The mixtures obtained by condensing glycerol with mixtures of ethylene oxide (20–80 parts) and propylene oxide (80–20 parts) so that there are at least 8 oxyalkylene groups per glycerol nucleus are useful as textile assistants, thickening agents, etc. C. O. C.

Starch Sizes and Finishes. C. M. Keyworth. *BP* 699,188

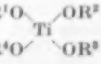
The viscosity of starch paste is decreased and its keeping qualities enhanced by adding the ester of a polyglycol or polyglycerol with a monobasic fatty acid. C. O. C.

Foaming Agents from Starch. Naamlooze Vennootschap W. A. Scholten's Chemische Fabriken. *BP* 699,530

Introduction by etherification and/or esterification of both hydrophilic and hydrophobic groups, the latter being of > 2 C, into starchy materials yields excellent foaming agents. C. O. C.

Solvent-soluble Water-repellency Compositions. DuP. *USP* 2,628,170–1

Addition of an ortho ester of titanium of formula



(some of the R's = Alk of < 7 C, the remaining R's = radicals of a 2-(lower alkyl)-1:3-alkane-diol in which the alkane group has a chain of > 3 C and the alkyl is of < 5 C) to a solution of wax in an organic solvent imparts to fabrics treated with the solution a much greater water-repellent effect than either the wax alone or a wax-aluminium soap mixture. After application removal of the solvent by normal drying suffices, there being no need for drying at high temperature. C. O. C.

Aqueous Dispersions of Cellulose Glycollic Acid or Carboxymethylhydroxyethyl Cellulose for producing Water-insoluble Coatings or Films. American Machine & Foundry Co. *BP* 698,948

An aqueous solution of a cellulose glycollate is acidified and the resulting freshly precipitated, highly hydrated cellulose glycollic acid, after being freed from excess precipitating acid and its salt, is homogenised or dispersed to yield a uniform, stable, smooth, gelatinous paste which can be used as coatings, sizes or adhesive or for forming into colourless, transparent, self-supporting continuous sheets or webs which after drying are completely water-insoluble, incapable of being rehydrated or swollen by water and incapable of being re-dispersed in water. C. O. C.

Fungicide. Scientific Oil Compounding Co. *BP* 698,971

The products obtained by reacting a phenol with a water-insoluble metal quinolinolate when dissolved or dispersed in an organic solvent are useful as fungicidal and/or fungistatic agents for textiles, leather, paper, etc. C. O. C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Steric Effects on Mesomerism. B. M. Wepster. *Rec. Trav. chim.*, **72**, 661–672 (Aug. 1953).

Phenol Synthesis by Direct Oxidation of Benzene. J. C. Chu, H. C. Al, and D. F. Othmer. *Ind. Eng. Chem.*, **45**, 1266–1272 (June 1953).

Benzene may be oxidised directly to phenol, without interference from secondary reactions, by an electric discharge of 3000–4000 v. under an optimum pressure of 6–12 mm. of Hg. Within the range 100–340°c. the effect of temp. is not important. The kinetics of the reaction and the design of the necessary plant are fully discussed. The efficiency of the process is low, but modifications to the plant (which are listed) may increase it to a level which is sufficient for commercial interest. J. W. D.

β-Naphthol—Solubility and Diffusivity in Water. M. P. Moyle and M. Tyner. *Ind. Eng. Chem.*, **45**, 1794–1797 (Aug. 1953).

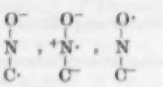
The solubility and diffusivity data of β-naphthol in water are presented for the temperature range 7–75°c. The current method for estimating liquid diffusion coefficients, when applied to this system, gave results that were 30–50% below the experimental values. A method is described for the determination of β-naphthol in low concentrations W. K. R.

Purification of *p*-Toluenesulphonyl Chloride. S. W. Pelletier. *Chem. and Ind.*, 1034 (26 Sept. 1953).

The *p*-toluenesulphonyl chloride (I) is dissolved in the min. amount of dry $CHCl_3$ and the resulting soln. diluted with 5 vol. of low-boiling petroleum ether. After separation of impurities by filtration the soln. is treated with an absorbent (Norit) and conc. to small vol., when (I) crystallises out. J. W. D.

New Reaction Between Aliphatic Hydroxylamines and Aromatic Diazonium Compounds. M. A. T. Rogers. *Chem. and Ind.*, 1033 (26 Sept. 1953).

The radical ion $R{\cdot}CH_2{\cdot}N(O^-){\cdot}\dot{C}HR$ (I) is unexpectedly stable and long-lived, and this may be attributed to resonance amongst the canonical forms—



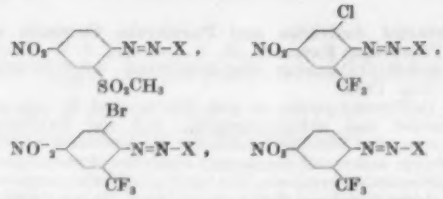
If this is so, other radicals may, in aq. alk. soln., attack either the C or the N atom. The decomp. of aryldiazonium salts in alk. reducing systems allows this hypothesis to be tested.

$$Ar{\cdot}N^+_2 + R{\cdot}CH_2{\cdot}N(O^-)CH_2{\cdot}R \longrightarrow Ar + N_2 + R{\cdot}CH_2{\cdot}N(O{\cdot}){\cdot}CH_2{\cdot}R \quad (II)$$

The radical (II) would then proceed, by inter- or intramolecular rearrangement, to the more stable radical anion (I). The expected end-products resulting from attack upon the C and N atoms (including those produced by further reactions such as autoxidation) have been confirmed experimentally with *p*-chloroaniline or aniline and diethylhydroxylamine, and with other aromatic amines and hydroxylamines. J. W. D.

Fluorine Substitution of Monoazo Dyes—Effect on Colour and Fastness Properties. J. B. Dickey, E. B. Towne, M. S. Bloom, G. J. Taylor, H. M. Hill, R. A. Corbitt, M. A. McCall, W. H. Moore and D. G. Hedberg. *Ind. Eng. Chem.*, **45**, 1730–1734 (Aug. 1953).

Fluorine substitution in either the diazonium or coupling component of monoazo acetate rayon dyes generally improves their fastness to light and to gas fading. Substitution in the *N*-(polyfluoroalkyl)aniline coupling component causes a change in hue from blue towards yellow depending on the number and position of the F atoms, but substitution in the diazonium component has little influence on hue. A series of orange, red and rubine dyes of excellent dyeing properties, and good fastness to light and gas fading is derived from the diazonium compounds—



and coupling components containing CF_2CH_2-, CHF_2CH_2-, $CH_2CF_2CH_2-$ or $CF_2CH_2CH_2$ groups. W. K. R.

Indicator and Dye Properties of Some Aromatic Phosphonic Acids and their Arsenic Analogues. G. M. Kosolapoff and G. G. Priest. *J. Amer. Chem. Soc.*, **75**, 4847–9 (5 Oct. 1953).

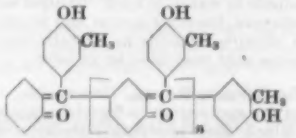
The phosphonic and arsenic analogues of Methyl Orange and Ethyl Orange show almost no affinity for cotton but good affinity for silk and wool on which they level well. The phosphonic and arsenic acid analogues of Congo Red have excellent affinity for cotton, wool and silk. The colour of the dyeings varies according to the pH of the fibre. The pH ranges of these dyes when used as indicators are given as well as the preparation of the dyes. C. O. C.

Spectrophotometric Determination of Ionisation Constants of Basic Dyes. S. Woislawski. *J. Amer. Chem. Soc.*, **75**, 5201–3 (5 Nov. 1953).

Spectrophotometric determination of the ionisation constants of 14 basic dyes of the azo, azine, xanthen, oxazine and thiazine classes in buffered 50% alcohol in 6 cases—Nile Blue A, Bismarck Brown Y, Rhodamine B, Chrysoidin Y, Neutral Red and Pyronine B—agreed fairly well with those obtained by titration in water and in 50% unbuffered alcohol. With the exception of Acridine Red they were the weaker bases of the dyes taken. With the other dyes spectrophotometric determination yielded a higher value in each case and these values paralleled the distribution ratios in buffer benzene. It seems that the ionisation constants vary with the method and medium and that the order of basicity is different in different media. C. O. C.

Oxidation of Phenols in Alkaline Solution—"Doctor Red". H. D. Taskis and G. E. Mapstone. *Chem. and Ind.*, 1061–1063 (3 Oct. 1953).

A purplish red colour develops in the doctor soln. used in refining shale gasoline, when it has been regenerated a few times; this colour was termed "doctor red". It was found that the colour was produced, upon alk. oxidation, only by phenols having an *o*-methyl group. Pure *o*-cresol yields no coloration, but when the brown contaminant resulting from oxidation during storage is present, the colour is formed—though the contaminant alone does not react. The rate of colour formation, the depth of colour, and its instability to further oxidation are increased when the phenol molecule contains tertiary butyl groups. A table is given in which the reaction colours and colour stabilities are listed for numerous pure and contaminated phenols; in some cases *o*-cresol is artificially contaminated with typical phenol oxidation products such as resorcinol, quinol, benzaldehyde, and benzyl alcohol. On a basis of the colour, mol. wt., and the response to various reagents, of phenolphthalein, aurine tricarboxylic acid, "doctor red", "*o*-cresol red", and "*o*-cresol brown", the following type structure is proposed for "*o*-cresol red"—



where *n* is any + ve integer, including 0. Some experimental details are given. J. W. D.

Photographic Colour-forming Development Reaction. W. A. Schmidt, V. Tulagin, J. A. Sprung, R. C. Gunther, R. F. Coles and D. E. Sargent. *Ind. Eng. Chem.*, **45**, 1726–1729 (Aug. 1953)

Coloured photographic images may be obtained by using a multilayer film in which suitable azine dye components are incorporated in the photographic emulsions.

When developed, these combine with the developer to form yellow, magenta, and cyan azine dyes. E.g. a benzoylacetanilide derivative, an 8-quinolinol derivative and a 6-anilino-1-naphthol-3-sulphonic acid derivative when developed with a 5-amino-2-arylamino-4-alkylaminobenzenesulphonic acid form yellow, magenta and cyan dyes respectively. The azine dye components have no detrimental effect on the photographic emulsions and simplicity in exposure and development is retained.

W. K. R.

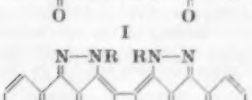
Some Adsorption Colours and Their Significance for Thermochromic and Tautomeric Effects. IV— Experiments with 1:3-Diketohydrindene Derivatives and with 1:4-Quinonedibenzenesulphonimides. A. Schönberg, A. Mustafa, and W. Asker. *J. Amer. Chem. Soc.*, **75**, 4645–7 (5 Oct. 1953).

When the colourless or yellow benzene solutions of several 1:3-diketohydrindene derivatives are treated at room temperatures with activated alumina, adsorption colours, believed to be caused by formation of the enolic forms, are produced almost immediately. 1:3-Diketo-2-phenyl-5-bromoindan exists in a pale yellow keto and a violet enolic form. The adsorption colour obtained is violet. Ethyl-benzoate solutions of this substance and indone show reversible thermochromic properties probably because the relative concentrations of the keto and the enol forms depend on temperature. The yellow 2-carbethoxy-1:3-diketohydrindene is considered to be the corresponding aci-ester. *p*-quinone and 1:4-naphthoquinonedibenzenesulphonimides also give adsorption colour on alumina.

C. O. C.

Vat Dyes of Pyrazoloanthrone Series. IV— *N*-Alkyl Derivatives of Pyrazoloanthrone Yellow. T. Maki and T. Akematsu. *Bull. Chem. Soc. Japan.*, **26**, 327–329 (Aug. 1952).

N-Alkyl derivatives of the two isomers of Pyrazoloanthrone Yellow (I and II, R = H) have been prepared, where R = *n*-propyl and R = *n*-butyl, by alkylation with the corresponding alkyl *p*-toluene-sulphonate. In both cases, and also when R = methyl or ethyl, the 9:9′- isomers (I) are rubine red dyes of high light fastness (5–6) and the 1:1′ isomers (II) are orange dyes of lower light fastness. Reflectance spectra of the four red dyes are given.



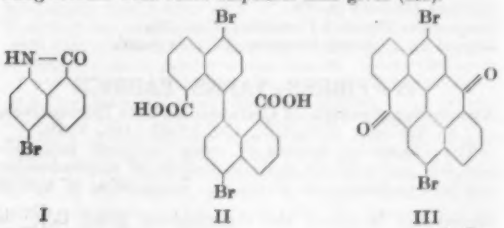
I

II

A. J.

Vat Dyes of Acenaphthene Series. V— Constitution of Dibromoanthanthrone (Indanthrene Brilliant Orange RK). T. Maki and N. Hashimoto. *Bull. Chem. Soc. Japan.*, **26**, 348–351 (Aug. 1953).

Indanthrene Brilliant Orange RK is shown to be 4:10-dibromoanthanthrone (III) by the following synthesis. Bromination of naphthostyril gives the 5-bromo derivative (I). Hydrolysis of the styril ring followed by diazotisation of the amino group and treatment with $CuSO_4$ and Cu gives the dibromo-dinaphthyl-dicarboxylic acid (II). Ring closure with conc. sulphuric acid gives (III).



I II III

A. J.

Leuco-cyanidin and Epicatechin. W. G. C. Forsythe. *Nature*, **172**, 726–727 (17 Oct. 1953).

A leuco-cyanidin isolated from cacao beans, gives chiefly a reddish-brown ppt. and some cyanidin and other polyphenols on heating with 10% HCl, as reported by Bate-Smith and Swain (*Chem. and Ind.*, 377 (1953)). Leuco-cyanidin with 0·1 N HCl gives mainly a reddish-brown ppt. (phlobaphene) and (—)epicatechin, which with boiling water is partly isomerised to (—)catechin. 1% oxalic acid and 0·1 N acetic acid behave similarly to 0·1 N HCl. It is considered that a close relationship exists between (—)epicatechin and part of the leuco-anthocyanidin molecule. The formation of cyanidin in strong acid is probably a side reaction.

A. J.

Colouring Material in Annatto—Butter Colour. S. Iversen and J. Lam. *Z. Lebensm.-Unters. u. -Forsch.*, **97**, 1–7 (1953): *Chem. Abs.*, **47**, 10146 (10 Oct. 1953).

Spectral absorption measurements of freshly extracted annatto, the same after heating and commercial butter colours indicated presence of various amounts of bixin (B). Probably heat transforms B as follows—

Cis-B ⇗ trans-B

B, (red) → B_2 (reddish orange) →

B_2 (orange) → B_3 (yellow) ..... colourless.

Commercial butter colours contain various transformation products of bixin. The transformations seem to involve one or more double bonds.

C. O. C.

Some Dyes of the Ancient South American Civilisations. G. A. Fester. *Isis*, **44**, 13–16 (1953): *Chem. Abs.*, **47**, 10232 (10 Oct. 1953).

Chemical and spectroscopic tests reveal that in Paracas in the 7th century B.C., three dyes and the natural brown colour of the wool were used to obtain a wide range of colours. The blue was indigo obtained from *Indigofera suffructicosa*, the yellow was on an alum mordant and has no characteristic spectrum. The red was also used on an alum mordant to obtain good carmines and was obtained from *Relbunium tetragonum* of the Sierra of Cordoba. It contains purpurin and pseudopurpurin. Cochineal from *Dactylopus coccus* was used in the Nazca and Chimu periods and must have been cultivated in the Inca period. To determine which red dye was used the fabric was boiled for a few seconds with NaOH, immediately acidified, extracted with butanol, the extract centrifuged and extracted with dil. NaOH, and the absorption measured. Cochineal absorbs at 571, 528 and 495 mμ, the purpurins absorb at 545, 505, and 280 mμ.

C. O. C.

Peacock Blue. A. S. Hester and C. H. Allen. *Ind. Eng. Chem.*, **45**, 1610–1618 (Aug. 1953).

A detailed description is given of the commercial preparation of Peacock Blue, a pigment derived from acid glaucine blue (C.I.671), which is widely used in printing inks. The dye is prepared in lake form by precipitation on aluminium hydroxide with the aid of barium chloride, and after filtration the wet pulp is "flushed" by kneading with linseed oil or other suitable vehicle, whereby the pigment is dispersed in the vehicle and the water is eliminated in one operation.

W. K. R.

Dispersible Pigment Powders. W. Carr. *Paint*, **23**, 363–5, 379 (Nov. 1953).

There is a growing tendency in many trades to use organic pigments as aqueous pastes rather than as dry powders, e.g. in dyeing paper there is a steadily growing demand for colours combining brightness with good fastness to light and this demand can be adequately met only by use of pigments. The use of pigments as powders generally results in dyeings in which large particles of undispersed pigments are present resulting in the full colour value of the pigments not being obtained. Pastes however have several drawbacks especially as regards storage, transport and handling so that attention is being paid to dispersible pigment powders. Addition of a dispersing agent can give a readily dispersible product, but as at least the same weight of dispersing agent must be used as pigment the cost is great in addition to which when the pigment paste is being dried after wet grinding with the dispersing agent, drying is often difficult and there are drawbacks to their use in paper coatings and dyeing and in emulsion paints. Products are available in which

the pigment is mixed with either a carbohydrate or an inorganic powder. These work satisfactorily in paper dyeing but are less satisfactory for paper coating and emulsion paints. They wet out readily in water; presumably because of the good solubility of the carbohydrate or inorganic compound but the degree of suspension is not very good though sufficient for paper dyeing where the beating action improves the dispersion. They wet out readily in polyvinyl acetate emulsions but even after high speed stirring are not well enough dispersed to give satisfactory brush-outs. Added to this most organic pigments cause thickening and breakdown of polyvinyl acetate emulsions if more than will give only a very pale tint is used. C. O. C.

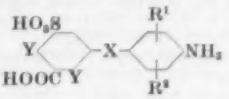
Report on Pigment Progress 1952-3. W. M. Morgans. *Paint*, **23**, 369–372, 379 (Nov. 1953).

A review of the literature including patents published during the year, 64 references. During the year there has been increased work in the development of more-readily dispersible pigments for use in either orthodox or other paint vehicles. The increasing demand for emulsion paints has emphasised the need for a wider range of pigments easily dispersible in water. There has also been considerable attention paid to the very fine grinding of pigments and extenders. C. O. C.

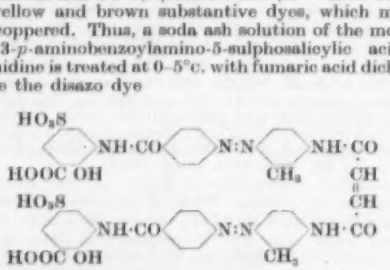
PATENTS

Copperable Polyazo Direct Cotton Yellows and Browns. S. *BP* 698,008

Two mol. of an azo compound



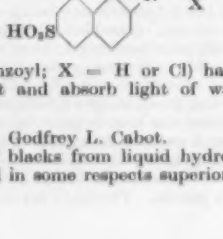
(X = –NH·CO⟨⟩N:N–, –NH·CO·NH⟨⟩N:N–, or –N:N–; Y = same or different, H or OH; R^1 = H, Alk, or OAlk; R^2 = H, Alk, OAlk, –NH·COAlk, or –NH·COOAlk) are linked together by reacting with 1 mol. of a dihalide Hal·CO·C(A):CH·CO·Hal (A = H or CH_3), to give yellow and brown substantive dyes, which may be after-coppered. Thus, a soda ash solution of the monoazo dye 3-*p*-aminobenzoylamino-5-sulphosalicylic acid → *m*-toluidine is treated at 0–5°C. with fumaric acid dichloride to give the disazo dye



which dyes cotton fast to light yellows which may be aftertreated with copper salts. E. S.

Nuclear Sulphonated 1-Carboxy-3-monocyclicaryl-benzo[f]quinoline Compounds. Kodak. *BP* 698,995

The products obtained by heating a monocyclic arylaldehyde, pyruvic acid and a 2-naphthylamine sulphonic acid in presence of water are useful as light filter dyes, e.g. water soluble salts of compounds of formula—



(R = H or benzoyl; X = H or Cl) have outstanding fastness to light and absorb light of wavelength 360–400 mμ. C. O. C.

Carbon Black. Godfrey L. Cabot. *BP* 699,406

Production of blacks from liquid hydrocarbons which are equal to and in some respects superior to gas blacks. C. O. C.

V—PAINTS; ENAMELS; INKS

Manufacture of Paints without Previous Drying of the Pigments. J. Sevestre. *Peintures, pigments, resins*, **29**, 495–7 (1953): *Chem. Abs.*, **47**, 10241 (10 Oct. 1953).

A review of the manufacture of flushed colours and their inherent economies. The process may be simplified by adjusting the wetting agents, the water content of pigment, and the type and amount of binder so that all but 1% of water in the paste is displaced. Pastes of Zn and Pb chromates, Prussian and Brilliant Blues, Toluidine, Para and Helio Reds were dispersed in this way in linseed oil, stand oils, oil varnish, alkyd and phenolic resins, and cellulose nitrate. Laboratory trials should precede setting up of displacement procedures as some pigments, e.g. Toluidine Red, may not require a wetting agent. C. O. C.

Fastness to Light of Printing Ink Pigments. T. E. Ludwig and S. R. Benson. *Amer. Ink Maker*, **31**, (2), 34–7 (1953): *Chem. Abs.*, **47**, 10242 (10 Oct. 1953).

Numerous red, blue, yellow, orange and green pigments were made into printing inks of three concentrations, prints of which were exposed in a Fadeometer. Tables are given showing the ink formulæ used and the light fastness of each pigment. C. O. C.

PATENTS

Pigment Dispersions. Postans. *BP* 699,311

The pigment is mixed with a carrier liquid of low viscosity and preferably volatile and is then subjected to ultrasonic vibrations. This results in a uniform dispersion of the pigment. The heat produced is used to evaporate part of the liquid so as to produce a preparation of the desired solids content. The product may be stored in bulk and is used for preparing paints. C. O. C.

Rapid Setting Printing Inks. ICI. *BP* 699,513

An ink which is very rapid setting, does not "mist" or spray when used on high speed presses and can be modified by addition of numerous solvents, resins, etc. comprises a medium or short chain length drying oil modified alkyd resin, a phenol-formaldehyde resin modified by an acidic natural resin and a polyhydroalcohol, and an organic solvent of b.p. > 200°C. C. O. C.

Coatings and Inks for Application to Polyethylene. S. A. Lookomsky. *USP* 2,628,208

A mixture of a dispersion of one resin and a solution of another resin is used. The first resin is compatible with polyethylene and soluble at high temperature in the higher boiling of the two liquids and the second resin is incompatible with polyethylene, insoluble at any temperature in the higher boiling of the two solvents but soluble in the lower boiling solvent at a temperature below that at which the first resin becomes soluble in the other solvent. C. O. C.

Coatings and Inks for Application to Polyethylene, Cellulose Acetate, Vinyl Resins etc. Emhart Manufacturing Co. *USP* 2,628,172

A dispersion or solution of polyethylene in a mixture of a solvent for polyethylene and a non solvent for polyethylene when coated or printed on to polyethylene, cellulose acetate or vinyl resin surfaces yields a strongly adherent coating or print. C. O. C.

Ultramarine for Colouring Vitreous Enamels. British Thomson-Houston Co. *BP* 699,625

Ultramarine is a satisfactory blue pigment for low temperature glazing vitreous enamels free from lead but containing sufficient TiO_2 to produce a light diffusing effect when the enamel is applied to glass. C. O. C.

Peacock Blue (IV p. 29).
Dispersible Pigment Powders (IV p. 29).
Report on Pigment Progress (IV this page).

VI—FIBRES; YARNS; FABRICS

Absorption Spectra of Cellulose and its Derivatives. E. Treiber. *Kolloid-Z.*, **130**, 39–63 (Jan. 1953).

The absorption spectra of many cellulosic materials and the methods for their experimental determination and interpretation are given. An explanation of optical degradation is given: the chromophore group C⟨O–⟩O– is

responsible for the weak absorption below 3000 mm^{-1} of cellulose which, on absorbing short wave light having a photon energy of 90 k.cal/mole, is ruptured at the glucosidic bond. From experiments on β- and γ-celluloses it is concluded that these are not merely fractions of different degrees of polymerisation but are also more or less chemically altered; some effects must have their origin in the C=O chromophore, probably due to uronic acids in the latter. Other, more technical applications of absorptiometry are discussed in detail. L. P.

Rayon, Heterogeneous Structure of. III. S. Okajima S. Hayama, and K. Watanabe. *Bull. Chem. Soc. Japan*, **26**, 322–327 (Aug. 1953).

Viscose rayon, cuprammonium rayon and a tyre cord are nitrated for varying times in a mixture of nitric acid, acetic anhydride and CCl_4. Cuprammonium increases in weight at a constant rate on nitration. With viscose and the tyre cord the rate increases suddenly after an initial slow reaction. On removing the nitrated skin with acetone and measuring the intrinsic double refraction (I') of the cellulose core, cuprammonium and the tyre cord give slightly decreasing values of I' with increasing thickness of the nitrated layer. Viscose shows a sharp decrease in I' at ca. 5% nitration. The results agree with those obtained by acetylation (Okajima, *et al*, *Ibid*, **25**, 271–275 (1952)). A. J.

Long X-Ray Spacings in Synthetic Fibres. K. Hess and H. Kiessig. *Kolloid-Z.*, **130**, 10–19 (Jan. 1953).

In almost all synthetic fibres long X-ray spacings occur due to periodicity of the ordered and disordered regions. The length of the spacing is the sum of those of the two regions. A summary is given of previous determinations of such spacings for many different fibres. They all lie between 70 and 200 A. and do not correspond to any integral multiple of the chemical repeat unit. A superlattice is thus formed which causes the small-angle scattering. By annealing the fibres the long spacings are increased. Swelling in phenol solutions does the same but the effect is partially reversed on washing out the phenol. The X-ray reflections show a pronounced broadening along the layer lines which is decreased by annealing.

Samples of Perlon L similar to single crystals were obtained by rolling stretched bristles. These showed slight broadening of the layer line when the beam was perpendicular to the rolled plane but strong broadening when parallel. The ordered regions are, therefore, ribbon-like.

Experiments with monochromatic beams proved that the usual band of filtered X-rays does not falsify the diagram. A complete analysis shows that the intensity of the long-spacing reflections of the superlattice is relatively higher than that of the atomic lattice. L. P.

Structure of Macromolecular Substances. I— Change in Length of Polyamide Filaments in Aqueous–Phenolic Medium. II— Diffusion Coefficient for an Aqueous–Phenolic Medium. E. K. Mankash and A. B. Pakshver. *J. Appl. Chem. U.S.S.R.*, **24**, 830–834, 835–839 (Aug. 1953).

The authors have previously studied the kinetics of the hydrolysis of polyamide fibres by acids (see *Chem. Abs.*, **46**, 7774 (25 Aug. 1952)) and have found that diffusion through the fibre and chemical reaction are rate-determining processes of about equal significance, the kinetics being adequately described by the equation of Rotinyan and Drozdov (see *Chem. Abs.*, **44**, 416 (26 Jan. 1950)). They now extend a previous investigation into the dimensional changes of polyamide fibres in aqueous–phenolic media by a study of the kinetics of changes in length (extension of undrawn fibres and shrinkage of highly drawn fibres), the conditions being similar to those used previously (see J.S.D.C., **67**, 473 (Nov. 1951)), and they find that the much slower diffusion of phenol as compared with that of the hydroxonium ion results in the dominance of diffusion as rate-determining process. Under the action of phenol, the macromolecules undergo rapid relaxation and slow disorientation, the latter process being so slow that a state of apparent equilibrium arises, which is attained throughout the fibre in 10–15 min. The kinetics follow the equation devised by Vulis (see *Chem. Abs.*, **35**, 5673 (20 Aug. 1941)) for the combustion of coal, a system in which the reacting gas has to diffuse through a constantly increasing crust of ash. For the present case the equation takes the form—

$$\frac{l}{l_0} = k'\sqrt{t} = k\sqrt{\frac{t}{Z}}$$

(l = change in length in time t (sec.), l_0 = ultimate change in length, Z = draft ratio) and is found to be obeyed closely for $l/l_0 < 0{\cdot}6$–$0{\cdot}7$ (Z is introduced in order to provide a const. k that is independent of the surface area of the fibre and is therefore proportional to the diffusion coeff.). A further function S, defined as L_0–l_0 where L_0 is the value of l_0 for the undrawn fibre (see J.S.D.C., **67**, 285 (July 1951)), is a measure of the departure of the form of the macromolecule from its most probable, folded form in the swollen fibre. With increase in draft, S rises and k falls (straightening and compacting of chains). With increase in phenol concn., S rises and k rises (weakening in intermolecular forces and facilitation of diffusion), and the difference in behaviour between drawn and undrawn fibres tends to disappear. With increase in temp., S remains unchanged, but k falls (the structure compacts without change in the form of the macromolecule). The value of S has a comparative significance only, for experiments on fibres that have been heated in the relaxed condition indicate that the undrawn fibre must in fact be regarded as partially drawn.

The Vulis relation may be derived independently from the equations for dye diffusion obtained by Crank (J.S.D.C., **66**, 366–374 (July 1950)), which lead, in the case of diffusing substances of low substantivity, to the value of

$$k' = \frac{4}{r}\sqrt{\frac{D}{\pi}}$$

(r = radius of fibre, D = diffusion coeff.), and from experiments similar to those already described and including determinations on fibres that have been previously heated in the taut condition, D is determined over a range of temp., thus permitting an estimate of the activation energy of the diffusion process. The results are closely in accord with those already cited; in particular, at a given temp. D varies over a wide range (1–400×10^{-10} sq.cm./sec.); it falls rapidly as the draft increases to $\sim 20\%$ and then only slowly, and preheating of the undrawn fibre in the taut condition, particularly at $> 150°$c., also leads to a great fall in D (increase in molecular interaction). A. E. S.

PATENTS

Non-woven Rayon Fabric. Minnesota Mining & Manufacturing Co. *USP* 2,262,883

A base layer of staple viscose and staple plasticised acetate rayon is bonded by heat and pressure to a warp of non-plasticised continuous acetate rayon filaments. The warp has a large number of ends per inch resulting in the final fabric being flexible and having one side having high surface sheen contrasting with the soft, lustreless, fibrous appearance of the other side. C. O. C.

Rapid Acetylation of Impregnated Cellulose (XI p. 35).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Enzyme Action in De-sizing. H. Appleyard. *Dyer*, **109**, 685–687 (15 May, 1953).

The liquefying action of enzymes on starch products, the relative importance of concentration, pH control, temperature, washing off and liquor to goods ratio, are discussed. There is a description of a continuous method of desizing which involves the use of J boxes in series. A. H.

VIII— DYEING

Application of Dyes to Textile Fibres at High Temperatures. C. L. Zimmerman. *Amer. Dyestuff Rep.*, **42**, P 627–P 634 (14 Sept. 1953).

A useful increase in the temp. of a dyebath is most readily obtained by means of an increase in pressure. At constant temp. an increase in pressure up to 50 lb. per in.^{2} has no effect upon dye penetration or levelness, or upon colour value. Various laboratory- and plant-scale machines are described by means of which cloth or yarn may be

dyed at temp. > 212°F. A semi-commercial continuous dyeing machine of 62" width has been developed by the Bachmann Uxbridge Worsted Corporation in which the pressure-dyeing chamber is preceded by a padder, the pressure (pneumatic) upon the rolls of which is arranged to be > that on the sealing rolls through which the material passes on entering the pressure chamber; these latter will allow 50 lb. per in.² pressure to be maintained whilst the cloth is in motion. Batch dyeing machines such as the Barotor (DuP) provide for the dyeing of small yardages which would not be economic for continuous dyeing; one of these, the Smith–Drum machine, is illustrated. Most machines of this type operate at or below 250°F. (continuous dyeing plant may operate up to 300°F.), above which level the advantage is too small to offset the high cost of equipment which will withstand the higher pressure. At high temp. dye molecule aggregates are broken down and fibre penetration by diffusion along inter-micellar spaces is facilitated. The improving effect of high temp. dyeing upon penetration, levelness, colour strength, and dyebath exhaustion is discussed, and there are comparative photomicrographs of cross-sections of fibres of Dacron (polyester), Orlon (acrylic), wool, nylon, cotton, viscose, Aralac, acetate rayon, and natural silk, dyed at both 210°F. and 290°F. Dacron (= Terylene) is best dyed at high temp., and under these conditions the dyeings have improved light fastness. With Orlon the use of assistants such as aniline or *m*-cresol may be obviated, and dyebath exhaustion is greater. Fibres are not, with the exception of acetate rayon, damaged at these high temp. providing the time of immersion is short. The thermal decomp. of many acid and direct dyes held at high temp. for several hours has been studied (there are three references to the literature) and the behaviour of 17 dyes is listed in the paper. J. W. D.

Dyeing of Textile Fibres under Static Pressure. L. Drijvers. *Teintex*, **18**, 354–369 (15 June 1953).

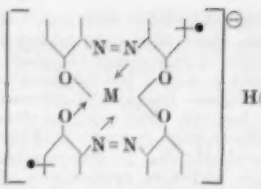
105°C. is given as the max. temp. to be used in dyeing wool, appreciable degradation of this fibre taking place at 115°C. and upwards. Dyeing is carried out in 3 stages: (1) Wool brought to the pH which will give complete exhaustion of the dye used at 105°C. (2) Raise to 105°C. within 15–20 min. (3) Dye for 5–10 min. at 105°C. The following pHs are given: Acid levelling dyes, pH 4–5; acid milling dyes (Polar and Cloth Fast type) pH 6; premetallised dyes (Cibalan and Irgalan type) pH 6 and Neolan type pH 2; chrome dyes pH 5–6; fast acid (Xylene P type) pH 4–5. With cellulosic fibres, decompn. of certain direct dyes is likely at high temps. and is avoided by adjusting the dyebath to pH 7 with ammonium sulphate. 110°C. is usually sufficient for Class A dyes but 125°C. is necessary for Class C direct dyes. B. K.

Influence of Preparation and Method of Dyeing and Finishing on the Colour and Colour Fastness of Vat-dyed Cottons. L. H. Bailey and F. J. Rizzo. *Amer. Dyestuff Rep.*, **42**, P 492–P 516 (3 Aug. 1953).

A 9 oz. sateen fabric is prepared for dyeing by 14 routines differing mainly in boil-off and degree of mercerisation, and dyed a standard shade by 4 common methods—pad-jig, pad-steam, Williams, and continuous booster. Some fabrics are treated with 3 different water repellent compounds comprising two cationic and one resinous. The dyed materials are evaluated for initial reflectance and for colour fastness after mobile launderings (including normal and with added chlorine), scrub tests, and outdoor exposures. Data obtained confirm that, in general, boil-off and mercerising lead to good colour fastness. The best dye method to be used depends on the preparation of the cloth and, in general, the pad-jig method gives the highest fastness. Theoretical aspects are discussed and it is emphasised that the best methods, both of preparation and dyeing, are those which provide the highest dye-fibre bonding strength and which do not produce a too rapid "strike" and consequent over-saturation of the fibre surface. Water repellent compounds protect the colour during wet mechanical operations. J. W. B.

New Methods in Wool Dyeing. H. R. Hirsbrunner, *Textil-Praxis*, **8**, 501–508 (June 1953).

The historical development of the premetallised dyes is discussed leading up to the production of the Irgalan (Gy) range. These dyes are unsulphonated azo cpds. containing 2 mol. dye : 1 mol. metal of the following general formula—



They are applied at pH 6–7 and reference is made to work indicating minimum abrasion resistance of wool on processing to lie in a pH range of 5–7. Rate of dyeing and migration curves for 11 Irgalan dyes are given and apart from insufficient fastness to potting and cross-dyeing they are all of high standard of fastness. B. K.

The pH Dependence of the Adsorption Isotherm and Adsorption Spectrum of Methyl Orange Bound to Human and Bovine Serum Albumin. J. R. Colvin. *Canadian J. Chem.*, **31**, 734–745 (Aug. 1953).

The difference between adsorption isotherms and adsorption spectra of methyl orange bound to human and bovine albumin at pH 6–8, 9·1, and 11·0 is studied. Results are discussed in terms of a limited reversible expansion of the human protein molecules, without unfolding, due to intramolecular electrostatic repulsion between groups. J. W. B.

Dyeing Properties and Usages of Synthetic Fibres. L. W. Müller. *Textil Praxis*, **8**, 783–790 (Sept. 1953).

The dyeing properties of polyamide, polyester, acrylic and vinyl polymer fibres are reviewed and the application of these fibres in the textile industry is indicated. If those composed of monofilament/multifilament polyamide fibres are dyed with suitable disperse dyes at 90–95°C. the monofilament fibre dyes appreciably heavier than the multifilament fibre, but solid shades are often obtainable by dyeing at 65–70°C. Anthraquinone vat dyes are applicable to polyamide fibres at temperatures nearing the boil and over-reduction of dye is prevented by the addition of cpds. to the dyebath such as sodium nitrite, cyanamide, glucose etc. The use of cyanamide is covered by the patented Calcotherm process. B. K.

Dyeing and Tannage. C. Faure. *Bull. assoc. franç. chim. inds. cuir. et doc. sci. et tech. inds. cuir*, **14**, 35–44 (1952): *Chem. Abs.*, **47**, 10261 (10 Oct. 1953).

The affinity of dyes for leather depends mainly on the changes in the leather and the dye ions though the structure of the dye also plays a part. Syntan leather has very little affinity for most acid dyes, but the affinity increases as the number of sulpho groups per dye mol. decreases and some triphenylmethane dyes dye syntan leather because of their amphoteric character. The electropositive character of chrome leather retanned with a syntan is restored by treating with colourless cationic compounds, e.g. Fixanol. Penetration depends on the mol. wt. of the dye and the number of sulpho groups it contains as well as on the sign of the charges. C. O. C.

PATENTS

Simultaneously Vat Dyeing and Dimensionally Stabilising Regenerated Cellulose Textiles. American Viscose Corpn. *USP* 2,628,151

The material is impregnated with the dye, a water-insoluble, alkali-soluble cellulose ether and formaldehyde at pH 1·2–6·5, dried in absence of contact pressure and preferably under tension, heat cured and, if necessary the dye developed. C. O. C.

Azoic Dyeing of Nylon and Acetate. General Aniline. *BP* 699,194

The material is impregnated with a coupling component and an amine base in presence of 1–5% (on wt. of fibre) of a guanidine compound after which an azo dye is formed on the material from the two components. The guanidine compound increases the affinity of the coupling component for the fibres, imparts greater stability to the diazonium compound and exerts a buffer effect which yields more even and brilliant dyeings of satisfactory depth and excellent fastness. C. O. C.

Dyeing Linear Polyesters. ICI. *BP* 698,490

The rate of dyeing is much increased if it is carried out at >100°C. and under superatmospheric pressure. C. O. C.

Dyeing Acrylonitrile Polymers with Azoic Dyes. Societe Rhodiaceta. *BP* 698,302

The material is treated in a bath which contains both the base and the coupling component and then in a second bath containing nitrous acid. The first bath is at 80–100°c. Intense colorations are obtained even on non-swollen material. C. O. C.

Dyeing Modified Polyacrylonitrile Staple Fibre Fabric. DuP. *USP* 2,628,152

Fabric made from fibres of copolymers of acrylonitrile and vinyl pyridines or vinyl picolines (*USP* 2,491,471) are dyed with acid dyes without danger of formation of rope and winch marks if 5–20 g./l. of a phenyl carboxylic acid, e.g. benzoic or salicylic acid, is added to the dyebath. This addition also enables dyeing to be carried out at 180–200°c. C. O. C.

Some Dyes of the Ancient South American Civilisations (IV p. 29).
Dispersible Pigment Powders (IV p. 29).

IX—PRINTING

Daniel Koechlin. 1785-1871. E. J. Mueller. *Teintex*, **18**, 369–373 (15 June 1953).

Life and work of Daniel Koechlin with special reference to various styles in calico printing. B. K.

Camile Koechlin. 1811-1890. *Teintex*, **18**, 567–571 (15 Sept. 1953).

Life and work of Camile Koechlin, son of Daniel Koechlin, with special reference to discharging of azoic dyeings and colour discharges on indigo. B. K.

Status and Problems of Colour Photography. J. Eggert. *Z. Elektrochem*, **52**, 712–716 (1952): *Chem. Abs.*, **47**, 9829 (10 Oct. 1953).

After a discussion of the additive and subtractive processes the following are listed as the main problems of modern colour photography— increase in speed of the emulsions, control of gradation within the different layers, image sharpness, improved colour contrast by masking techniques, and better stability of the dyes forming the image. The colour films now available and their important characteristics are compared. C. O. C.

PATENTS

Printing Basic Dyes on Cellulosic Textiles. C.P.A. *BP* 699,086

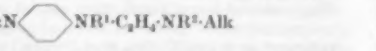
Modification of *BP* 566,258 (J.S.D.C., **61**, 135 (1945)). Aqueous solutions of basic dye and a salt of a lake-forming acid, e.g. phosphomolybdic acid, are mixed in presence of a buffer to keep the pH of the mixture at 5·0–12·5. This mixture is then printed on and acidification effected after steaming by means of an acid not weaker than formic acid. C. O. C.

Photographic Materials Sensitive to Light of High Intensity and Short Duration. Typon AG für Photographische Industrie. *BP* 699,307

A finely divided impact-detonatable, endothermic, *N*-containing compound, e.g. nitrogen iodide, is incorporated into a fibrous absorbent base, e.g. cloth or filter paper. The product is stable and safe to handle. It is sensitive to the light produced by electronic flash units and yields immediately on exposure direct positives of good definition and full density range. The nitrogen iodide image is readily converted into a silver image by treating for 5 min. in alcohol, then for 10 min. in 5% aq. $AgNO_3$, rinsing, treating in 10% aq. $Na_2S_2O_3$ until the brownish-black coloration is removed and finally treating in alkaline 5% aq. sodium stannite. C. O. C.

Diazotype Prints. Kalle & Co. *BP* 699,112

The light sensitive layer used contains as the azo coupling component 2:3-dihydroxynaphthalene-6-sulphonic acid and as the light sensitive substance a water-soluble diazo compound containing the group



(R^1 = Alk, aralkyl or $-C_2H_4{\cdot}N(C_2H_5)_2$; R^2 = Alk or Ar; the benzene group may be further substituted). The dark blue prints obtained by use of this layer are not sensitive to acid. C. O. C.

Diazotype Process for Producing Photomechanical Printing Plates. Kalle & Co. *BP* 699,412

The light sensitive constituent in the plate consists of a water-insoluble ester or subst. or unsubst. amide of a sulphonic or carboxylic acid of 2-diazonaphthol(1) or 1-diazonaphthol(2). The plate is exposed to light under an original and then developed with an alkaline solution to remove the light-affected diazo compound. The plate may then be rubbed with a greasy ink. After use the plate may be cleansed with an alkaline detergent. *BP* 699,413

The exposed plate is developed so that the diazo compound unacted upon by light forms an azo dye with an azo coupling component. C. O. C.

Developing Electrostatic Images. Battelle Development Corpn. *BP* 698,994

A latent electrostatic image on an insulating layer has a conductive electrode placed within 0·5 in. of it after which a charged finely divided material is introduced between them so that it becomes deposited on the insulating layer in close conformity with the latent image. C. O. C.

X—SIZING AND FINISHING

Acrylic Resins in Textile Processing. A. C. Nuessle and B. B. Kine. *Ind. Eng. Chem.*, **45**, 1287–1293 (June 1953).

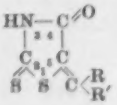
The chemical constitution and physical properties of acrylic resins are briefly discussed, the latter in particular connection with stiffness and the temp. of transition from the glassy to the rubbery state. Method of deposition can also affect physical properties, e.g. a film laid down from an emulsion may be less continuous than one laid from solvent soln. Acrylic polymers may be applied (1) from aq. soln., (2) from solvent soln., (3) from emulsion, or (4) as monomer, with subsequent polymerisation *in situ*. A range of fabric "handle" may be obtained with acrylic resins applied from emulsion, and fabrics may be endowed with stiffness or softness, or given a full, heavy "handle". With cellulosic materials the adhesion of polymer to fibre is invariably good, and the type of finish is controlled by the character of the polymer itself. Methyl methacrylate adheres well, but has a high fusion point; its inter-particle cohesion is thus poor, and it may be used to delustre a shiny fabric. Drying temp. influences the cohesion of polymer particles and thus affects the quality of the finish significantly; it is of little importance when a polymer is deposited from solvent soln. The fusion of a hard polymer may be aided by the presence of plasticiser, and a stiff finish obtains. A polymer applied to delustre a fabric must not be used in presence of a softer polymer which will plasticise it, otherwise stiffening will occur instead of dulling. Acrylic polymers which adhere sufficiently well may be used as yarn sizes; their application to cotton is very limited, but with nylon, polyacrylic acid is of considerable value. Polymethacrylic acid is less effective, and possible reasons for this are suggested and discussed at length. Polyacrylic acid may be left in the woven nylon to act as a durable finishing agent when the nylon is heat-set at ca. 200°c. For a crisp organdie finish, e.g. on a nylon lace, the polyacrylic acid is best applied after weaving so that both warp and weft yarns are sized. In presence of glycerol an even crisper finish is obtained, and this strongly resists removal by acid or alk. soln. Adequate adhesion is not obtained with Orlon or Dacron. The alkali salts of polyacrylic and polymethacrylic acids give aq. soln. of high viscosity and can be used as thickeners, e.g. in printing pastes; the corresponding amides behave similarly, and they may be insolubilised by the action of H·CHO or CHO·CHO to impart durable crispness, glazing, embossing effects and resistance to shrinkage. A variety of effects including shrinkage resistance, altered texture, decreased water imbibition, and greater resistance to abrasion, can be obtained upon several fibres including wool, viscose, and cotton, by the application of an acrylic or vinyl monomer which is then polymerised *in situ* after the catalyst has been introduced in a separate operation. Cotton reacts directly with acrylonitrile in presence of NaOH to give cyanoethyl cellulose. Many of these effects are illustrated by experimental data given in ten tables. J. W. D.

Mildew-preventing Activity of Rhodanine Derivatives—(I) Ketone Derivatives. F. C. Brown, C. K. Bradsher, and E. N. Lawton; **(II) Some 5-Arylidene Derivatives.** F. C. Brown, C. K. Bradsher, and S. M. Bond. *Ind. Eng. Chem.*, **45**, 1027–1030, 1030–1033 (May 1953).

(I) Many fungicides in general use are organic sulphur compounds containing the grouping—

(1)

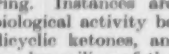
Rhodanine also contains this grouping, and though it has been patented as a fungicide its protective action is limited. Twenty-five compounds resulting from the reaction of rhodanine with ketones, and in which the above toxiphoric grouping (1) is preserved, have been examined for mildew-proofing activity; they have the structure—



(2)

Treated strips of cotton fabric were subjected to pure culture tests using *Chætomium globosum*, and to soil burial tests, details of which are given. After exposure the strips were examined for visible mildew growth, and subjected to tensile test. Results are tabulated for aliphatic and alicyclic ketone derivatives, and for a number of 3:5-disubstituted rhodanine derivatives. The protective activity of aliphatic ketone derivatives in which R′ in (2) is CH_2 rises to a max. when R = amyl, followed by a sharp decline when R = hexyl. This pattern of activity is paralleled by alkylated phenols and resorcinols, and by aliphatic bases. Some of the alicyclic ketone derivatives give good protection against mildew, the extent of which depends upon the number of C atoms attached to the alicyclic ring, and upon their position with respect to the rhodanine ring. Instances are cited of a strong resemblance in biological activity between certain of the derivatives of alicyclic ketones, and those of aliphatic ketones which can, by coiling of their C chains, produce the same skeletal configuration of C atoms. A table showing the performance of a number of other fungicides under the same test conditions is given as a basis of comparison.

(II) Rhodanine reacts readily with aromatic aldehydes, and 31 simple benzylidene rhodanines (30 containing substituents in the aryl grouping), and 4 products of the condensation of acetophenone with rhodanine, have been evaluated as fungicides and rot-proofing agents. The toxiphoric groupings (1) above, and—



are both present in these compounds. 5-Benzylidene rhodanine itself has, unlike simple rhodanine, a marked protective action. The activity of the substituted derivatives depends on the nature, number, and position of the substituent groups; the results are presented in 7 tables, and are fully discussed. The fungistatic activity of 5-benzylidene rhodanine may be markedly improved by the presence of substituent groups, but a tertiary amino group so introduced may completely nullify its activity. The most active of these derivatives are those in which the substituents are halogen or alkyl groups. The results do not confirm the theory that these compounds act by virtue of their ability to precipitate certain essential trace metals from the medium. J. W. D.

PATENTS

Raising the Melting Point of Polyamide Fibres. Inventa A.G. für Forschung und Patentverwertung. *BP* 698,337–8

Treatment with the vapour of an aldehyde at 120–180°c. in absence of water, swelling agent or catalyst considerably raises the m.p. without causing loss in strength. C. O. C.

Single-ply Corrugated Fabric. United States Rubber Co. *USP* 2,627,644

A plain open weave fabric the warp of which consists of alternate rows of heat shrinkable yarns and non-heat shrinkable yarns is heated under conditions whereby the fabric is provided with evenly spaced transverse curves or undulations. This produces a lightweight corrugated fabric having pronounced resistance to compression. C. O. C.

Linen Finish on Cotton Fabrics. Heberlein & Co. *BP* 699,559

The dry fabric is treated for 10–30 sec. in NaOH 10–20°Bé. at < 0°c., washed, neutralised and washed. The process can be carried out in mercerising, uses less NaOH than previous methods and there is less tendency for the fabric to shrink. C. O. C.

Resin Finish. William Clark & Sons. *BP* 698,244–5

Cloth, especially linen interlining, is given increased resistance to creasing and improved dimensional stability by impregnating it with neutralised glue or gelatin or a solution or suspension of a protein or starch, drying, impregnating with a precondensate of a synthetic resin, drying and baking. **C. O. C.**

Water-repellent Finish. Th. Goldschmidt. *BP* 699,210

When treating textiles with organosilicon halides to render them waterproof damage by the halogen hydride formed is avoided if the moisture content of the textile is first reduced to < 3%. C. O. C.

Fire-resistant Cloth. Titan Co. *BP* 698,742

The cloth is impregnated with an aqueous solution of a tetravalent titanium halide capable of forming a transparent gel, the solution in the cloth gelatinised, treated with an alkali and finally washed and dried. Preferably the solution also contains antimony chloride. The treated cloth is flame and glow resistant and is little altered in appearance or texture. The flameproof is fast to laundering. C. O. C.

Bonding Cellulose Yarns to Rubber. Courtaulds. *BP* 699,649

Incorporation in the fibres of 3–6% by wt. of a compound of formula—

$$R(NH{\cdot}CS{\cdot}S{\cdot}M)_n$$

(R = hydrocarbon radical of valency *n*; *n* = 2, 3 or 4; M = Monovalent metal or basic radical) results in better bonding. C. O. C.

Water-repellent Finish. Union Carbide & Carbon Corpn. *BP* 699,479

Textiles are rendered water-repellent by treatment for 5 min. at 60–65°c. in a bath containing 0·7–2·0% of a composition made up of wax (35–70 parts by weight), a dissociable metal salt, e.g. basic aluminium sulphate or zirconyl oxychloride (0·5–2·0), an organic amine (0·5–2·0), polyvinyl alcohol of saponification value 0–20 (1–20), followed by drying at 120°c. C. O. C.

Coating Flexible Bases with Polyvinyl Chloride Compositions. Gevaert. *BP* 698,266

The plasticised polymer is heated and then ground into powder. This powder is spread on cloth or other flexible base and bonded thereto by heat and pressure and finally heated at a still higher temperature to strengthen the coated material. C. O. C.

Coated Fabric. DuP. *BP* 699,049

A coating which does not penetrate the fabric is obtained by applying a mixture of (a) ungelatinised polyvinyl chloride dispersed in a plasticiser which is a non-solvent at room temperature but which dissolves the polymer on heating, and (b) a solution or dispersion of a polyvinyl acetate resin in a volatile liquid which does not dissolve polyvinyl chloride. The plasticiser used must be a plasticiser for both the polymers. The coated fabric is then heated to evaporate the solvent, further heated to gelatinise the polyvinyl chloride and finally cooled. C. O. C.

Surface Decoration of Polyvinyl Chloride Sheets or Coatings. B.X. Plastics. *BP* 699,683

The material is embossed, then coated with a "plastisol", i.e. suspension or paste of a vinyl chloride polymer or copolymer in a plasticiser, and finally heated so that the embossing is at least partially released and the plastisol gelled. The product has an embossed pattern which is unaffected by reheating and is more resistant to wear than the original embossed sheet or fabric. C. O. C.

Bias Binding. Broadstones Synthetics. *BP* 698,251

Cloth is cut on the bias into strips on to each of which latex is extruded in strips lengthwise and is then heated to gel the latex. If necessary the cloth may be impregnated with latex before the strips of latex are applied. C. O. C.

Simultaneously Vat Dyeing and Dimensionally Stabilising Regenerated Cellulose Textiles (VIII p. 32).

Rapid Acetylation of Impregnated Cellulose (XI below).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Adsorption of Methyl Cellulose on Polystyrene. O. Deile, H. Krämer and W. Klaus. *Kolloid-Z.* **130**, 105–110 (Feb. 1953).

The refractive index of sols. of polystyrene with added methyl cellulose is a good indicator of the state of peptisation. In the region of greatest flocculation, i.e. when the weight ratio is 1 : 20, the refractive index reaches a minimum and there is a steep drop in the charge of the particles as measured by their electrophoretic mobility. The phenomena are attributed to adsorption of the methyl cellulose. L. P.

Rapid Acetylation of Impregnated Cellulose. J. C. Thomas. *J. Amer. Chem. Soc.*, **75**, 5346–5350 (5 Nov. 1953).

Cellulose impregnated with certain aqueous solutions and dried is readily and rapidly acted upon at $> 90°$c. by acetic anhydride with formation of soluble, high mol. wt. cellulose triacetates. Thus cotton linters impregnated with urea (38% on wt. of cellulose) and $(NH_4)_2SO_4$ (2) reacted with excess acetic anhydride in 1 min. at 138°c. to yield a soluble triacetate with a DP (degree of polymerisation) of 585. In the same system at 95°c., acetylation was complete after 13 min. to yield a product with DP720. Other compounds can replace urea and ammonium sulphate. It is necessary to have two impregnating agents, one, e.g. $(NH_4)_2SO_4$ to act as a catalyst and the other to swell the cellulose though there is some evidence that acetylation of the swelling agent is a necessary part of the process. C. O. C.

PATENTS

Sized Felted Vegetable Web Material. ICI. *BP* 698,334

A process for the production from vegetable fibre of an aqueous suspension thereof, suitable for making felted vegetable fibre web material, comprises forming a suspension of the fibre in water containing a water-soluble aluminium salt, a water-soluble resin soap, a sequestering agent (for preventing precipitation of aluminium soaps from the solution), and deacetylated karaya gum. The gum may be added after the fibres are beaten in the solution stated. The aqueous suspension may contain dispersed rosin, and the sequestering agent may be a water-soluble sodium salt of a phosphorus oxyacid of the phosphorus pentoxide stage of oxidation, e.g. sodium hexametaphosphate, tetrasodium pyrophosphate or sodium tripolyphosphate. The sequestering agent may be one capable, when dissolved in water, of giving aminopolyacetate ions in which each nitrogen atom attached to an acetate radicle is a tertiary nitrogen atom linked to not less than two such radicles. These aminopolyacetate ions may be derived from nitrilotriacetic acid, or ethylene diamine tetracetic acid or their mono- or disodium or ammonium salts. S. V. S.

Imprinting Coatings on Paper and Paper Board. Consolidated Water Power & Paper Co. *BP* 698,648

Aqueous liquid, e.g. a plasticisable water-borne substance, is continuously applied to the surface of the web and the composite is plastically worked between rolls to form a smooth plastically worked surface to which a thin film of water-borne mineral coating material is applied, and immediately afterwards another film of water-borne mineral coating material is imposed continuously under pressure by means of a coated roll and the coated web dried continuously and calendered. The coated product thus obtained has a greater surface smoothness and brightness than a product similarly plastically worked and then coated similarly with a single layer of thickness equal to the combined thickness of the two films. The first film applied is doctored immediately to render it smooth and thin, before perceptible drying occurs, and all the steps in the process may be carried out without fully drying between steps. S. V. S.

Aqueous Dispersions of Cellulose Glycollic Acid or Carboxymethylhydroxyethyl Cellulose for producing Water-insoluble Coatings or Films (III, p. 27).

Dispersible Pigment Powders (IV p. 29).

Coating Flexible Bases with Polyvinyl Chloride Compositions (X p. 34).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Polar Amino Acid Content of Collagen and Related Material. J. Cassel, E. McKenna, and A. Glime. *J. Amer. Leather Chem. Assocn.*, **48**, 277–288 (May 1953).

The acidic amino acids of hide powder are determined by two chromatographic procedures, and the glutamic acid content of collagen, hide powder, and gelatin are estimated by conversion to pyrrolidone carboxylic acid. Acidic and basic amino acid analyses are made on hide powders which have been modified by deamination, methylation, formaldehyde tanning, and by treatment with trypsin, and on a chrome tanned leather. No significant differences are found in the amino acid analyses of collagen, gelatin, or hide powder; that of hide powder is not altered by chrome or vegetable tanning or by treatment with trypsin. Deamination for one week reduces the lysine by 80% and prolonged deamination reduces the arginine value. Methylation considerably reduces the aspartic acid value of hide powder. J. W. B.

Studies on the Reactive Groups of Collagen. III—Cross-linkage and its Relation to Tanning. R. L. Sykes. *J. Soc. Leather Trades Chem.*, **37**, 294–303 (Sept. 1953).

Collagen in the form of pelt strips is treated with a number of reagents to introduce cross-links. Mercuric acetate, and other group 2b. acetates are used as one group of compounds; epichlorhydrin and two other 1,2 epoxides as the other. An apparatus is devised for recording heat-shrinkage temperature, which is used as a measure of cross-link stabilisation. Such treated collagen has enhanced hydrothermal stability, the products being leather-like in character; this is suggested as evidence for a theory of tanning involving the introduction of stable cross-linkings between adjacent polypeptide chains. J. W. B.

Reaction of Vegetable-tanned Leather. I—With Formaldehyde. S. S. Kremen. *J. Amer. Leather Chem. Assocn.*, **48**, 333–343 (June 1953).

Formaldehyde may be used for the post-tannage of leather prior to, during, or following the bleaching process, and a wide range of reaction conditions is available. When applied to vegetable tanned insole leather the process leads to greatly increased resistance to deterioration, though it is of comparatively little value for outer leather since this requires mechanical improvement rather than chemical stability. The degree of tannage is rather higher under acidic conditions than under alkaline, but in the latter case the shrinkage temp. is markedly higher. Under certain conditions the presence of the borate ion is beneficial, but there appears to be a range of pH outside which it may even be deleterious. The presence of NaCl has little effect under acidic conditions, but appears to lead to a higher degree of tannage in alkaline media; this may be largely due to tannin insolubilisation. There is evidence that the HCHO combines with both the hide substance and the vegetable tannins simultaneously. The experimental methods are described and the results presented in tabular form. J. W. D.

Tanning with Melamine and Formaldehyde. W. Windus. *J. Amer. Leather Chem. Assocn.*, **48**, 513–516 (Aug. 1953).

Methylolmelamines have been used for some time in tanning, but a method is described whereby the necessity for pre-condensation of the melamine and the HCHO is eliminated, both being added to the tanning soln. in the free state. It is suggested that there is reaction of the

Mannich type between the methylol groups of the trimethylolmelamine which is formed in soln., and the amino groups of the collagen. Active H atoms are supplied by the melamine and this obviates the necessity for a strong acid. It is essential to maintain a fairly low ratio of HCHO to melamine, or to restrict the amount of melamine used, otherwise the formation of a three-dimensional polymer, and the high degree of cross-linking with the collagen, result in a leather of low tensile strength. J. W. D.

Dyeing and Tannage (VIII p. 32).

XIII—RUBBER; RESINS; PLASTICS

Finishes for Glass Fabrics for Reinforcing Polyester Plastics. L. P. Biefield and T. E. Philipps. *Ind. Eng. Chem.*, **45**, 1281–1286 (June 1953).

Glass fabric for use in polyester laminates, after cleaning by heat treatment at ca. 650°C., may be treated with a bonding agent such as an organosilicon compound such as vinyltrichlorosilane, diallyldiethoxysilane, a siloxanol salt, or vinylsiloxane, or an organochromium compound such as methylacrylatochromic chloride. There is probably reaction between the organosilicon compounds and the glass surface, siloxane linkages being formed; the allyl or vinyl groups react with the polyester. The temp. necessary for the fixation of the various organosilicon bonding agents are tabulated. Methods are described for the evaluation of the finishes. J. W. D.

PATENTS

Preventing Discoloration by Hydrogen Sulphide of Vinyl Chloride containing a Cadmium Stabiliser. Firestone Tire & Rubber Co. *BP* 699,193

Vinyl chloride compositions containing 0·5–5·0% (on wt. of polyvinyl chloride) of an organic cadmium compound as stabiliser is protected against discoloration by H_2S by adding 0·5–10·0% of an aminoplast condensate or resin. C. O. C.

Plasticisers for Polyvinyl Acetate. Deutsche Gold- und Silber-Scheideanstalt. *BP* 699,113

The esterification product of a butylene glycol, preferably 1:4-butylene glycol, and a C_4–C_4 mixture of fast running fatty acids is a good plasticiser for polyvinyl acetate. C. O. C.

Pigmented Compositions for Colouring Plastics, Rubber, Waxes, Gums, etc. B. F. Goodrich Co. *BP* 699,185

Colour master batches of extremely high quality are obtained by milling a pigment with a solid plastic in presence of a liquid dispersion and grinding medium which can subsequently be recovered and reused. Thus the liquid can consist of water and a water-miscible solvent for the resin but must not be such as to dissolve the plastic or permit it to gel or agglomerate. C. O. C.

Embossing Thermoplastic Film. M. A. Chavannes. *BP* 699,060

The film is applied to a carrier, heated and embossed while hot. This enables the embossing period to be brief and the film and carrier to be readily cooled thus even when working at high speeds the film and the carrier may be sufficiently cooled as they pass around the embossing roller to cause the embossed design to be permanent and sharp. C. O. C.

Surface Decoration of Polyvinyl Chloride Sheets or Coatings (X p. 34).

XIV—ANALYSIS; TESTING; APPARATUS

Practical Way for the Separation of Coloured Liquids. D. H. Kohn. *Chem. and Ind.*, 1053 (3 Oct. 1953).

The separation of two deeply coloured liquids whose mutual boundary is difficultly visible may be facilitated by adding a few drops of a soln. of β-methylumbelliferone, and then illuminating the separating funnel with ultra-violet radiation. J. W. D.

Colorimetric Determination of Calcium with Ammonium Purpurate. M. B. Williams and J. H. Moser. *Anal. Chem.*, **25**, 1414–1417 (Sept. 1953).

The use of a high pH (pH 11·3) greatly increases the sensitivity of the method (formerly described only as applicable to macro quantities of Ca) and permits its extension to the micro-range; errors due to the decomp. of the dye in alk. soln. are minimised, the reliability of the method being thus enhanced. The effect of various anions and cations on the absorbancy is discussed; Ba, Sr, Mg, and Li also form compounds with ammonium purpurate, and Fe^{2+} and Hg^{+} may be tolerated only up to 1 mg./l. The curve obtained in presence of the Cl^- ion does not differ significantly in slope from that obtained with NO_3^-, but that obtained with SO_4^{2-} does so. Experimental details and absorption spectra are given. J. W. D.

Determination of Nitrate in Sodium Nitrite. C. L. Johnson. *Anal. Chem.* **25**, 1276–1277 (Aug. 1953).

An aq. soln. of the sample is added gradually to cold conc. H_2SO_4 in an evacuated bottle (of which there is a diagram), under which conditions the side-reaction—

$$3HNO_2 = HNO_3 + 2NO + H_2O$$

which occurs as a result of local high concn. of nitrite, is gradually reversed when it does take place. The nitrite is decomp. as—

$$HNO_2 + H_2SO_4 = HNOSO_4 + H_2O$$

The NO_2^- is titrated with standard $FeSO_4$ soln. Full details, and results of trial analyses, are given. J. W. D.

Colorimetric Determination of Ammonia and Cyanate. J. M. Kruse and M. G. Mellon. *Anal. Chem.*, **25**, 1188–1192 (Aug. 1953).

The sample soln. is adjusted to ca. pH 7 by means of a sodium acetate–acetic acid buffer, a 3% soln. of chloramine-T is added, followed by a reagent (the preparation of which is described fully) containing pyridine, pyrazolone, and bispyrazolone. When colour development has taken place the soln. is extracted with CCl_4 and the absorbance of the extract measured spectrophotometrically at 450 mμ. The effect of many variables is discussed, and a list of interfering ions is given; Al, Co, Cu, Fe, Hg^{2+}, Ni, Ag, and Na interfere to different extents in the determination of NH_3, and CN^- and SCN^- in that of OCN^-. When NH_3 and OCN^- are simultaneously present, the former may be removed by means of an ion exchange column containing a sulphonic acid resin in the sodium salt form. It is not possible to separate OCN^- from CN^-, but a procedure is described by which they can be estimated when both are present. J. W. D.

Determination of D-Glucose in Corn Syrups by use of Glucose Dehydrogenase. R. L. Whistler, L. Hough, and J. W. Hylin. *Anal. Chem.*, **25**, 1215–1216 (Aug. 1953).

D-Glucose may be estimated in the presence of other sugars by enzymatic oxidation with glucose dehydrogenase to D-gluconolactone, which is titrated with standard alkali. The method is fully described and the source of the enzyme is indicated. J. W. D.

Infrared Spectra for Analysis of Aldehyde and Ketone 2:4-Dinitrophenylhydrazones. J. H. Ross. *Anal. Chem.*, **25**, 1288–1303 (Sept. 1953).

The infrared spectra of the 2:4-dinitrophenylhydrazones of aldehydes and ketones are highly characteristic, and permit the easy identification of compounds which differ only by $-CH_2-$, or whose 2:4-dinitrophenylhydrazones have closely similar m.p. As little as 10 mg. of derivative may be clearly identified in less than 30 min. Spectral curves and data are given for 25 aldehydes and ketones including HCHO, CH_3CHO, acetone, mesityl oxide, glyoxal, crotonaldehyde, and benzaldehyde, and these are discussed. Different polymorphic cryst. forms of the 2:4-dinitrophenylhydrazones yield different infrared spectra, but this introduces no difficulty into the procedure. J. W. D.

Anodic Voltammetry of Phenols. J. F. Hedenburg and H. Freiser. *Anal. Chem.*, **25**, 1355–1358 (Sept. 1953).

Phenol can be oxidised at a micro Pt anode, yielding a current–voltage curve similar to the conventional polarographic wave. A reproducible half-wave potential is obtained which is a function of the pH; at pH 9 it has a value of 0·52 v. An equation is derived which describes this dependence. A polarographically reversible one-electron oxidation to the phenoxide free radical is indicated by the experimental evidence. The diffusion current is closely proportional to the concn., and essentially independent of pH. Phenols of different half-wave potential may be identified by this means; mixtures may be resolved only if the individual half-wave potentials are $\ll$ 0·2 v.

apart. The method has some application in quant. analysis, but its accuracy is limited. J. W. D.

Separation of Phenols by Partition Chromatography. T. R. Sweeney and J. D. Bultman. *Anal. Chem.*, **25**, 1358–1363 (Sept. 1953).

Certain mixtures of phenols may be separated by partition chromatography using the system water (on silicic acid)–*iso*-octane, and the fractions examined spectrophotometrically at 230 mμ. The proportion of water to silicic acid in the system is critical with respect to the rate of movement of each phenol through the column and to the diffusion of each zone. Variation of the water–silicic acid ratio may enhance the resolution of a particular mixture of phenols. In general the concn. of phenol is not important, but at high concn. the resolution may be impaired by the increased spread of the bands. The order in which a group of compounds emerge from a column follows, in general, their partition coefficients; in accordance with this *o*-isomers have smaller R_f values than *p*- or *m*-isomers, and are thus easily separated from them. The size of the *o*-substituted group is of considerable importance, and by the choice of a suitable water–silicic acid ratio, mixtures of phenols containing *o*-substituents of various sizes may be resolved. Ten chromatograms are shown in illustration of the principles involved; one of these relates to the resolution of a phenolic fraction of creosote. J. W. D.

Ultraviolet Analysis of Isomeric Cresol Mixtures. G. E. Carney and J. K. Sanford. *Anal. Chem.*, **25**, 1417–1418 (Sept. 1953).

From Beer's law, at a given wave length, the absorbance (A) of a mixture of cresols is given by—

$$A = (a_o c_o + a_m c_m + a_p c_p)b$$

where a is the absorptivity in l./g.cm., c is the concn. of a constituent in g./l. of soln., and b is the optical path in cm. The subscripts denote the isomers. Where three isomers are present, measurements at three wave lengths, and three equations are required, but only two measurements and equations when only two specific isomers are known to be present. The optimum wave lengths for the cresols are—2728 mμ (meta), 2774 mμ (ortho), and 2858 mμ (para.). Care must be taken to ensure that phenol, and xylenols (and higher homologues) are absent since these interfere seriously. The analyses of five synthetic mixtures of isomers are reported and discussed; the results were satisfactory. J. W. D.

Microdetermination of *N*-1-Naphthylphthalamic Acid Residues in Plant Tissues. A. E. Smith and G. M. Stone. *Anal Chem.*, **25**, 1397–1399 (Sept. 1953).

N-1-Naphthylphthalamic (I) acid is hydrolysed with 30% NaOH, the amide linkage being severed to yield sodium phthalate and 1-naphthylamine; the latter is steam-distilled from the soln. and coupled with diazotised sulphanilic acid. The resultant red azo dye is determined spectrophotometrically at 534 mμ., a reagent blank being used for reference. The method, which is fully detailed, will detect as little as 2 μg. of (I), and gives 80–100% recovery in the 5-μg. range. There is a diagram of the apparatus. J. W. D.

Spectrophotometric Determination of Anthraquinone and Benzanthrone. K. B. Whetsel. *Anal. Chem.*, **25**, 1334–1337 (Sept. 1953).

Mixtures of anthraquinone (I) and benzanthrone (II) may be analysed by measuring the absorbance of their soln. in conc. H_2SO_4 at 266 and 515 mμ. (II) may be determined by the absorbancy at 515 mμ. without interference from either (I) or anthrone. The concn. of (I) and (II) may be calc. from the data—

$$\%(I) = 55{\cdot}07\ A_{266m\mu} - 41{\cdot}07\ A_{515m\mu}$$

$$\%(II) = 241{\cdot}0\ A_{515m\mu}\ (A = \text{corrected absorbance})$$

Experimental details are given, and results are discussed. The acid soln. are stable for several hours. J. W. D.

2:4:7-Trinitrofluorenone as a Reagent for Microscopic Fusion Analysis— Application to Polynuclear Aromatics and some Substituted Benzenes. D. E. Laskowski, D. G. Grabar, and W. C. McCrone. *Anal. Chem.*, **25**, 1400–1404 (Sept. 1953).

A simple microscope fitted with a Kofler hot stage is used to determine, in a single heating cycle, the m.p. of the unknown compound, of the 2:4:7-trinitrofluorenone derivative, and of the two eutectics formed between the derivative and, respectively, the unknown and the reagent. These data permit the clear identification of many polynuclear aromatic hydrocarbons and their derivatives. The four fusion temp., together with ancillary general information, are tabulated for 30 polynuclear compounds, and there are photomicrographs of the mol. compounds formed. The technique, fully detailed, must be followed closely in order to obtain the required accuracy. Three pairs of compounds (some of which are not strictly polynuclear aromatics) are not sufficiently differentiated by the method, these are— 1-bromonaphthalene and 1-iodonaphthalene, azobenzene and diphenyl, and *p*-aminoazobenzene and stilbene; simple supplementary tests such as colour and refractive index will differentiate between these pairs. The method appears to be applicable to condensed carbon and condensed heterocyclic systems and, by use of different reagents, to other categories, e.g. amines, using picric acid. J. W. D.

Paper Chromatographic Separation of Intermediates in Food Colours. Y. Hashimoto and S. Shimizu. *J. Pharm. Soc. Japan*, **73**, 767–9 (1953): *Chem. Abs.*, **47**, 10141 (10 Oct. 1953).

3:6:2-$(HO_3S)_2C_{16}H_5OH$ (I) and 6:8:2-$(HO_3S)_3C_{16}H_4OH$ (II) were isolated by a quantitative spot test with a round filter paper, and developed in a low-pressure, closed vessel with butanol, water and acetic acid (5:4:1). The separated zones were cut out and extracted, and colour was developed by alkaline coupling with diazonium solution of 4:1-$H_2NC_{10}H_6SO_3H$ as Amaranth (III) and New Coccine (IV). These red dyes were measured colorimetrically. I, II, III and IV were purified by the chromatopile from which pure products were obtained. This test is a rapid and simple way for both qualitative and quantitative analysis of coaltar dyes used for colouring foods. C. O. C.

Systematic Identification of Japanese Official Water-soluble Food Colours by Paper Chromatography. Y. Ishida, N. Inagaki, A. Shiota, and R. Watanabe. *J. Pharm. Soc. Japan*, **73**, 736–740 (1953): *Chem. Abs.*, **47**, 10141 (10 Oct. 1953).

Food colour, solvent, and R_f value (in order given) are listed for identification: Orange I, 10% HCl, 0·05; Guinea Green B, 10% HCl, 0·92; Erythrosin, 25% ethyl alcohol–5% ammonia (1 : 1) (I), 0·47; Eosin, I, 0·56; Rose Bengal, I, 0·60; Phloxin, I, 0·70; Ponceau 8X, 80% phenol (II), 0·25; Ponceau 3R, II, 0·45; Ponceau R, II, 0·45; Naphthol Yellow S, II, 0·52; tetrazine, phenol-ethanol-water (40:25:40) (III), 0·38; Amaranth, III, 0·48; Indigo Carmine, III, 0·52; New Coccine, III, 0·59; Sunset Yellow FCF, III, 0·60; Light Green SF Yellowish, III, 0·78–1·00; Fast Green FCF, III, 0·78–1·00; Brilliant Blue FCF, III, 0·78–1·00. R_f values depended on the mol. wt. and dissociation constant. C. O. C.

Analysis of Lacquer Thinners by Fluorescent Indicator Adsorption Method. W. H. Ellis and R. L. LeTourneau. *Anal. Chem.*, **25**, 1269–1270 (Aug. 1953).

A modification of the method of D. W. Criddle and R. L. LeTourneau (*Anal. Chem.*, **23**, 1620 (1951)), in which *n*-butylamine is used as the displacing agent, being applicable to lacquer thinners which already contain alcohols and other strongly adsorbed oxygenated compounds, and Rhodamine "B" Base (DuP) is added to the fluorescent indicator to mark the boundary between the oxygenated compounds and the *n*-butylamine. Other minor modifications are described, and the accuracy of the method is discussed on a basis of experimental evidence. J. W. D.

Determination of Nitrogen in Cellulose Nitrate and of Cellulose Nitrate and Combined Phthalate in Lacquers. W. E. Shaefer and W. W. Becker. *Anal. Chem.*, **25**, 1226–1231 (Aug. 1953).

The nitrate N content of nitrocellulose may be determined by dissolving the material in hot acetic acid, boiling with a reagent consisting of 2*N*-$FeCl_2$ containing HCl and HBr, and titrating the resulting Fe^{2+} with $TiCl_3$ after Knecht and Hibbert. The method may be applied directly to nitrocellulose lacquers without the necessity of separating the nitrocellulose (sometimes impossible), a 12% N content of the nitrocellulose being assumed in the calculation. Combined phthalate in any alkyd resin remaining after the treatment with $FeCl_2$ may be extracted with

methylene chloride and its phthalate content found by a modification of the Kappelmeier method (gravimetric determination as potassium phthalate). A further modification is required if diglycollic acid is present. Full details are given and there is a photograph of the apparatus. J. W. D.

Compression Extraction Method for Extracting Textile Materials. H. W. Wolf and R. S. Rankl. *Amer. Dyestuff Rep.*, **42**, 617–620 (14 Sept. 1953).

The weighed material, in the form of loose fibre, is contained in a vertical cylindrical brass vessel which tapers to a $\frac{1}{8}$ in. opening at the lower end, and compressed by means of a plunger which is loaded by a weighted lever. A measured amount of the extracting liquid (CCl_4) is introduced above the plunger, and seeps down through the compressed fibrous mass; the plunger fits very loosely into the extraction tube. The emergent liquid is collected in a light metal weighing dish and the solvent removed in a forced-draught oven. Data are presented in four tables for comparable extractions of oiled woollen stock, woollen yarn, dry combed top, and raw wool, by the Soxhlet and the compression extraction methods; the compression extraction procedure is of slightly less precision, but is quite adequate for routine determinations. The apparatus is shown both diagrammatically and in a photograph. J. W. D.

Improved Apparatus for the Quantitative Separation of Fibre Mixtures by Flotation. C. H. Giles and I. S. Shaw. *J. Textile Inst.*, **44**, T 406 (Aug./Sept. 1953).

The fibre flotation and separation device of Mhatre and Tawde (*J. Textile Inst.*, **44**, T 70 (1953)) is modified in such a way that mercury need not be used. A separating funnel dips into the flotation tube in such a manner that the stem extends well below the surface and, when fibre separation is complete, some of the heavier liquid is run in. This displaces the upper fibrous layer over through a side arm into a collecting tube. J. W. B.

Accuracy of Quantitative Paper Chromatography in Amino Acid Analysis—Addendum. R. C. Salander, M. Piano, and A. R. Patton. *Anal. Chem.*, **25**, 1252–1253 (Aug. 1953).

Further details regarding the accuracy and details of the experimental procedure described by A. R. Patton and P. Chism (*Anal. Chem.*, **23**, 1683 (1951)). J. W. D.

Microdetermination of Polyvinylpyrrolidone in Aqueous Solution and in Body Fluids. G. B. Levy and D. Fergus. *Anal. Chem.*, **25**, 1408–1410 (Sept. 1953).

The colorimetric method of W. R. Thrower and H. Campbell (*Lancet*, 1096 (1951)), based on the reaction of polyvinylpyrrolidone (I) with iodine, is severely limited in sensitivity, and the relationship between absorbance and concn. is non-linear. By a readjustment of the concn. to ensure a small molar excess of iodine, a linear relationship is obtained. The sensitivity is greatly improved by control of pH (with a citric acid buffer) and elimination of the partial pptn. of (I). The method is given in full, and data from trial analyses are reported and discussed. The absorbance varies slightly with the mol. wt. of (I), and though this effect is minimised by the relative consistency of commercial preparations of (I), for work of the highest accuracy the calibration curve corresponding to the K value of the sample must be used, if this is not done an error of 10–25% may result. The method is sensitive to 5–30 p.p.m. (I) in the final dilution, and this may be extended to 1 p.p.m. J. W. D.

Separation of Pigments and Fillers from Mineral Tanning Agents. L. Seligsberger. *J. Amer. Leather Chem. Assocn.*, **48**, 503–513 (Aug. 1953).

A critical study of existing methods of analysis (which is reported at length) leads to the proposal of a systematic method of analysis for all kinds of mineral leather, by means of which the mineral tanning agents may be determined without interference from inorganic pigments and fillers. The separation is best effected with an organic acid mixture. The procedure, which is described fully, covers the estimation of Al, Cr, Fe, Zr (in presence of Ti), and Zn, in the soln. resulting from the wet oxidation of the leather. J. W. D.

Determination of Zirconium in Tanning Materials and in Leather. J. Wendkos and I. C. Somerville. *J. Amer. Leather Chem. Assocn.*, **48**, 355–358 (June 1953).

Both Zr and Ti are pptd. from acid soln. by means of *p*-hydroxyphenylarsonic acid, and determined gravimetrically. The Ti is separately estimated by the acid H_2O_2 method, and the Zr thus found by difference. Full experimental details are given for the analysis of Zr tanning materials for Zr, Ti, free acid (as SO_3), and basicity (expressed according to Schorlemmer's system), and the analysis of Zr tanned leathers for total ash, mineral constituents (Zr, Ti, and Cr). J. W. D.

Temperature Effects in a Rotation Viscometer. C. C. Mill and E. R. Gates. *Anal. Chem.*, **25**, 1390–1393 (Sept. 1953).

The accumulation of frictional heat at high rates of shear is known to affect the validity of results obtained with rotational viscometers. The data recorded in an experiment may be simply corrected if the temp. coefficient of viscosity and the equilibrium temp. of the viscometer bob are known. The method applies both to simple oils and to pigmented suspensions; in the latter instance the hysteresis loop previously reported is shown to be due to temp. changes during the experiment. The results which are presented and discussed relate to mineral oils, and suspensions of carbon black in them. J. W. D.

Note on the Rapid Determination of Free Formaldehyde in Phenol-Formaldehyde Syrups. J. Haslam and W. W. Soppet. *J. Appl. Chem.*, **3**, 328–329 (July 1953).

The syrup is dissolved in 90% (v/v) ethanol and the soln. brought to neutrality as shown by bromophenol blue indicator. Hydroxylamine hydrochloride soln. is added in excess, and the soln. allowed to stand for at least $\frac{1}{2}$ hr. (if this is not observed, an accurate end-point cannot be obtained) before titration of the liberated HCl with N-NaOH soln. The sudden change in pH which represents the end-point occurs at pH 3·40. The experimental details are given in full. J. W. D.

Determination of Molar Ratio of Urea to Formaldehyde in their Condensation Products. P. P. Grad and R. J. Dunn. *Anal. Chem.*, **25**, 1211–1214 (Aug. 1953).

Existing methods for analysing urea-HCHO condensates are discussed and criticised. In the proposed method, the resin is split by H_3PO_4 and the HCHO which distils over is trapped in an excess of alk. KCN soln. The determination of HCHO is completed by precipitating excess CN^- as the Ag salt, which is purified and weighed. If halogen is present in the distillate it may be determined by the Volhard method, excess HCHO being added to prevent interference by any KCN which remains. This method may also be applied to the estimation of HCHO in melamine-HCHO resins. Urea is converted by reaction with benzylamine to dibenzylurea which, being highly insol., may be determined gravimetrically. The urea in fully cured resins cannot be wholly recovered by this method. The molar ratio of the components is calc. from the expression—

$$\frac{2 \times \%\text{HCHO}}{\%\text{Urea}} = \text{X} \qquad 1 : \text{X} = \text{urea} : \text{HCHO}$$

The method is given in full, its precision is discussed, and there is a diagram of the apparatus. J. W. D.

Observing the End-point in Tests of Latex Mechanical Stability. A. C. Meyer. *Anal. Chem.*, **25**, 1348–1350 (Sept. 1953).

The procedure for determining the end-point in the A.S.T.M. method for measuring latex stability is unsatisfactory and is subject to considerable error. It is recommended that a set of narrow 80-mesh Monel metal screens (flat mesh) should be introduced perpendicularly to the direction of flow of the liquid during the high-speed stirring, at suitable intervals. The coagulated particles thus filtered out at each stage give a clear indication of the type and extent of the breakdown, and the proportion of dispersed solids coagulated may be determined at any stage by weighing. The method has a significantly lower error than the A.S.T.M. procedure. There are photographs of two sets of screens dipped during the breakdown of latices, and these are discussed. J. W. D.

p-Anisidine, a Sensitive Reagent for Cupric Ions. A. Kh. Batalin. *J. Anal. Chem. U.S.S.R.*, **7**, 182–183 (May–June 1953).

A saturated aq. soln. (1 ml.) of *p*-anisidine is dissolved in 0·1 N-KCNS (100 ml.) and this reagent (2–3 drops) is added to the soln. under test (1 ml., neutral), when a red coloration is obtained in presence of Cu^{2+}, 0·6 μg. of Cu being detectable. The sensitivity of the reagent is unaffected by Mn^{2+}, Al^{3+}, Zn^{2+}, Mg^{2+}, Cd^{2+}, Ni^{2+}, Cr^{3+}, Fe^{2+} (at concn. up to 500 times that of Cu^{2+}); by Co^{2+}, Ag^{+}, Sn^{2+}, UO_2^{2+}, Ce^{3+}, Hg^{2+}, Pb^{2+} (up to 50 times); or by Hg_2^{2+} and Sb salts (up to 5 times). Interference of Fe^{3+} is prevented by addition of NaF. A. E. S.

Determination of Nitrate in Sodium Nitrite. C. L. Johnson. *Anal. Chem.*, **25**, 1276–1277 (Aug. 1953).

An aq. soln. of the sample is added gradually to cold conc. H_2SO_4 in an evacuated bottle (of which there is a diagram), under which conditions the side-reaction—

$$3HNO_2 = HNO_3 + 2NO + H_2O$$

which occurs as a result of local high concn. of nitrite, is gradually reversed when it does take place. The nitrite is decomp. as—

$$HNO_2 + H_2SO_4 = HNOSO_4 + H_2O$$

The NO_2^- is titrated with standard $FeSO_4$ soln. Full details, and results of trial analyses, are given. J. W. D.

Extraction of Water-soluble Dyes from Food. M. Mottier and M. Potterat. *Mitt. Gebiete Lebensm. u. Hyg.*, **44**, 293–302 (1953): *Chem. Abs.*, **47**, 10752 (25 Oct. 1953).

Methods for extracting water-soluble dyes with organic solvents are reviewed and a simple procedure using quinoline at room temperature is described. Pre-extraction with ethyl alcohol in a water bath was necessary in a few cases. Many dyes, including those used as food dyes in Switzerland, were examined and completely extracted with quinoline. Extraction and separation of the layer were facilitated by working in presence of a buffer solution of pH 3. The extracted dyes were identified chromatographically and by spot tests. C. O. C.

Dynamic Fatigue Test and its Evaluation. W. Wegener. *Melliand Textilber.*, **34**, 640–641 (July); 742–745 (Aug. 1953).

A special testing machine, in which the maximal strain decreases proportionally to the elongation, has been described (*Z. gesamte Textilind.*, **52**, 27, 55, 79, 108 (1950); *Melliand Textilber.*, **31**, 668 (1950); *Kautschuk und Gummi*, **4**, 57 (1951)). A process for evaluating test results and judging rayon qualities is developed. The total elongation (permanent and elastic) is determined as a function of time, and a relationship is established between the forces of the dynamic and those of the static fatigue test. For estimating the elastic behaviour, the efficiency (elastic extension : total extension) and the elasticity number (alteration in stress : alteration in extension) are introduced. Yarn of viscose filament rayon is shown to deform very rapidly at first, later only slightly, but before rupture, very great deformation occurs again. From a plot of the elasticity number against time, it can be seen that the strength of the yarn improves up to a point during the test. A more complete communication appears in *Z. gesamte Textilind.*, **54**, 10, 71, 105 (1952). H. E. N.

Apparatus for Testing Resistance to Bending. G. Schröder. *Melliand Textilber.*, **34**, 418 (May 1953).

Cf. *GP* 880,955. This "Flexotest" consists of a vibrating spring, released at a constant amplitude by an electromagnet. It is designed for material of low stiffness, which is bent backwards and forwards over its full length by the swings of the spring. The time required to dampen the vibrations to a certain degree, indicated by means of an optical system, is measured. The resistance to bending is given in terms of the damping effect in sec. A modified tester is being built. H. E. N.

Determination of Water Retention of Fibres and Yarns. A. Zart. *Melliand Textilber.*, **34**, 849 (Sept. 1953).

Detailed directions for the method employing centrifugation. H. E. N.

Determination of Moisture Regain of Spun Jute. E. Burlet. *Bull. Inst. Text. France*, (38), 21–29 (April 1953).

Although some volatilisation of fatty matter occurs when moisture evaporates from sized jute, results of satisfactory commercial accuracy are obtained when the sized fibres are dried for 1½ hr. at 110°C. G. L.

Determination of Traces of Heavy Metals in Cellulosic Materials. F. Pohl and E. Treiber. *Melliand Textilber.*, **34**, 747–748 (Aug. 1953).

Spectrometric determination is complicated particularly by the presence of relatively large quantities of Ti in delustred material. The method developed uses the substituted dithiocarbamate "T-Carbat" of Glen and Schwab (*Z. angew. Chem.*, **62**, 320 (1950)), which at pH 5 in acetic acid soln., containing tartrate, precipitates all the heavy metals of interest, but leaves Ti, Al, alkali and alkaline-earth metals in soln. Zn acetate is added to coprecipitate traces of the heavy metals. Full details are given. In 1 g. cellulose 0·00001% Cu, Fe, Mn; 0·0001% Co, Ni, V; 0·001% Cr, Pb, Mo, can be determined. Sodium diethyldithiocarbamate can be used but is less sensitive for Cr and Mo. H. E. N.

Detection and Measurement of Modification in Wool. M. Robinet. *Bull. Inst. Text. France*, (39), 49–60 (May 1953).

Samples of wool treated under various alkaline conditions were examined in several laboratories by means of standardised testing methods and the results critically compared. G. L.

Shrinkage of Fabrics made from Wool. M. van Overbeke, G. Mazingue, and P. Outreman. *Bull. Inst. Text. France*, (38), 7–20 (April 1953).

A comparative study of standardised testing methods applied to fabrics varying in weaving construction. G. L.

Physiology of the Perception of Surface Colours. E. N. Willmer. *J. Oil & Col. Chem. Assocn.*, **36**, 491–499 (Sept. 1953).

The structure of the human eye, and the mechanism of colour vision (so far as this is now known), are discussed. The retina, upon which the image falls, is composed of ~ 130,000,000 separate cells whose photosensitivity depends on the lability of certain carotenoid pigments. Visual acuity is at a max. near the optical axis, and as the periphery is approached colour values become inaccurate and perception of fine detail poor. Only ~ 1,000,000 nerve fibres connect the retina to the brain and there is thus considerable pooling of "information" *en route*. A detailed diagram shows this. The study of simple eyes such as those of *Limulus polyphemus* leads to the belief that the messages transmitted along optic nerves consist of impulses in the form of propagated variations in the electrical p.d. which normally exists across the surface membrane of a nerve fibre. In vertebrate animals the impulse patterns are complex, owing to the complex way in which "pooling" takes place in the retina. In a simple eye such as that of *Limulus* the receptors are all of the same kind, and hue and intensity cannot be observed simultaneously. When two types of receptor are present, forming a diodic system, hue discrimination is possible, but confusions are liable to occur when certain impulse differences between the two paths are reproduced in different ways, e.g. certain purples may be confused with certain greens; this occurs, with a normal human eye, in certain types of colour-blindness. The human eye appears to be triodic (at least), and the receptors are of at least three types, whose max. sensitivity is believed to be, respectively, in the yellow ($\lambda_{max.}$ = 575μ.), green ($\lambda_{max.}$ = 540μ.), and violet ($\lambda_{max.}$ = 450μ.) regions; for identification these are loosely termed the red, green, and blue receptors respectively. Colour sensations appear to depend mainly upon differences between receptor responses rather than upon the responses themselves, and intensity is believed to be judged by a separate pathway. The exact nature of the receptors themselves has not yet been established with certainty, but the blue receptors appear to be different in character from the other two.

Photoreceptors, like all sense organs, "adapt" themselves to conditions, and do not respond continuously with the same frequency to a continued constant stimulus. Prolonged exposure to light reduces the sensitivity of the

retinal receptors which are affected by the stimulus, at the same time possibly enhancing the sensitivity of those which are not stimulated. The longer a red surface is looked at the less sensitive become the red receptors and, to a lesser extent, the green; the blue receptors gain in sensitivity. If the gaze is diverted to a white surface this will probably appear to be blue–green—a purely subjective hue. Examples are discussed of the importance of this effect in schemes of decoration and lighting. In delicate colour matching it is advantageous to move the eyes and to scan the samples rather than to stare at them continuously in turn. The blue receptors recover from these adaptations at a quite different rate from the red and the green; they are also believed to contribute very little to the sensation of brightness as distinct from hue. The above effects apply to surfaces and fields of fairly large area, uniformly illuminated. A number of colour "illusions" associated with small-field colour distributions are described, e.g. a yellow spot surrounded by blue appears to be white when viewed at a distance. The precise cause of these effects is not known, but a contributory factor is believed to be chromatic aberration (for which the human eye is not corrected), and the described "illusions" are discussed in terms of this hypothesis. Another contributory factor in small-field colour effects is the adverse effect of the pooling of receptor responses; at the periphery of the eye, colour estimation is most inaccurate and pooling is at a max. The blue receptors are almost absent from the fovea, and when certain colours are fixated by this part of the retina (especially at close distances) this can lead to confusions, e.g. blue–green with orange; these are characteristic of tritanopic colour blindness—the fovea is essentially colour-blind, being diodic and tritanopic. Other colour effects are connected with the time sequence of events in the retina, and depend upon the difference in the time for reaction on the part of the receptors or pathways responsible for the appearance of the two colours. The perception of surface colours depends therefore not only upon the nature of the reflected light, but also upon many physiological factors concerning the structure and the state of adaptation of the human eye. J. W. D.

Colour Matching and Colour Measurement. T. Vickerstaff and I. S. M. Walls. *J. Oil & Col. Chem. Assocn.*, **36**, 507–530 (Sept. 1953).

The effect of illumination intensity on the accuracy and ease of colour matching has been studied. Seven trained colourists in turn assessed the order of intensity within three sets of patterns (two of wool, one of cellulose acetate mouldings) each representing ten closely graded steps in colour intensity. The room in which the assessments were made was arranged to secure max. diffuseness of lighting, and freedom from reflection of coloured light. Compact illumination was provided by 30 daylight fluorescent tubes (working at a colour temp. of 6500°K.) wired so that 1, 4, or all 30 tubes could be used at any one time—giving light intensities of 14, 50, and 330 ft.-candles respectively. The results, which are fully reported and discussed, were analysed statistically. Within the range 14–300 ft.-candles the illumination intensity has no significant effect on the acceptance or rejection of near colour matches. Since this illumination range includes all levels of intensity likely to be met in practice, there seems to be no possibility that an experienced observer would accept, under poor conditions of illumination, a match which he would reject under better conditions. In a further trial the observers were required to state which of each of eight pairs of patterns (arranged in two sets—red cellulose acetate, and green wool) was the bluer; the patterns were presented randomly so that each pair was examined three times, at each of three levels of illumination, by each of nine observers. The distribution of mistakes showed that accuracy improved with increasing intensity of illumination to a max. at 80–100 ft.-candles (about 8 of the tubes), above which level, there was no advantage in increasing the intensity. The level of accuracy was better with the red patterns than with the green ones. It was generally agreed that the best conditions were represented by an illumination of 60–80 ft.-candles at the working surface, and that the glare-free, diffuse lighting was advantageous.

A major difficulty in colour matching procedure is the verbal description of small differences in hue, strength, and brightness existing between closely similar patterns. Such terms as "bluer", "weaker" and "brighter" are commonly used, and their significance is generally agreed, but precise agreement can never be attained in the descriptions of the same small colour difference by a number of highly trained observers. The only wholly satisfactory solution would be to measure such differences objectively and numerically. Accepting the C.I.E. system as satisfactory for the description of colour differences, it was decided to ascertain whether a recording spectrophotometer might be used for the measurement of small colour differences of the order represented by normal commercial colour matching tolerances. Four sets of painted panels, normally used to test candidates for colourists' posts, were examined systematically using three different recording spectrophotometers; in two cases the curves were integrated by the thirty selected ordinate method to obtain the mean trichromatic values, whilst in the third a Librascope continuous integrator was used. The automatic integrator gave results of superior accuracy. The results are analysed, hue, strength and brightness being expressed and discussed in terms of the C.I.E. system. The same test panels were examined by six colourists, visually, and their verbal descriptions of the colour differences were correlated with the C.I.E. data; very good agreement was observed, only two cases of definite disagreement (both concerned with hue) were observed. The investigation thus shows that colour differences of the order of colour matching tolerances may be satisfactorily measured by a recording spectrophotometer, especially if associated with a continuous recording device such as the G.A.F. Librascope integrator. Colour measurements made on different spectrophotometers proved to be in excellent agreement. The lengthy discussion (covering 7 pages) following the presentation of this paper, and that by E. N. Willmer (*ibid.*) is reported fully. J. W. D.

Specifications for the Evaluation of the Colour Fastness of Textiles. P. Rabe. *Melliand Textilber.*, **34**, 649–650 (July 1953).

The historical development since 1951 is described. H. E. N.

Identification of Aminoplasts. E. Elöd and H. Jörder. *Melliand Textilber.*, **34**, 860–863 (Sept. 1953).

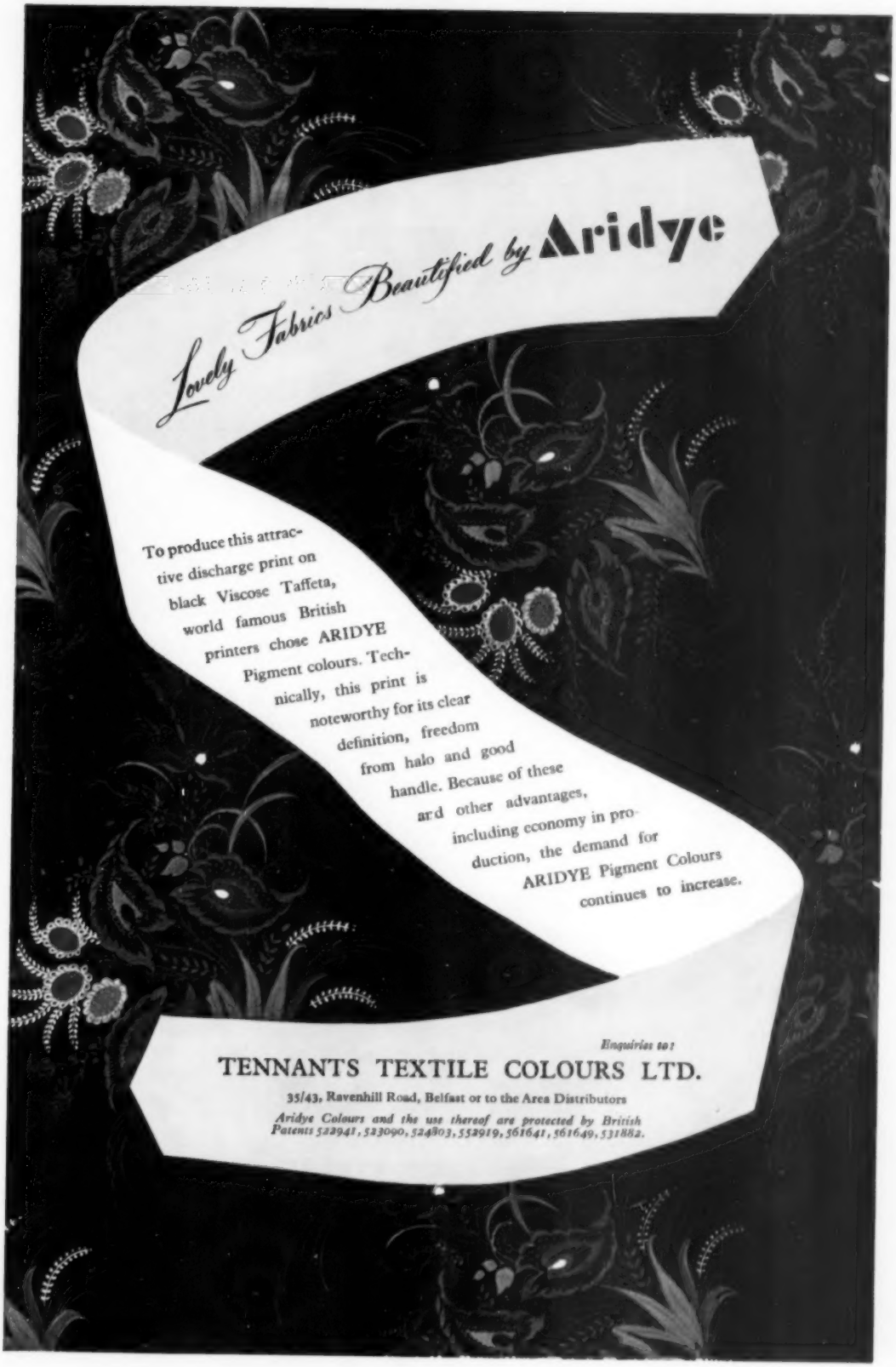
Formaldehyde is detected by its odour when a sample (3–4 sq.cm.) is treated with boiling 2% H_2SO_4 (5 c.c.). The vapours can be cooled and led into carbazole (0·004 g.) in H_2SO_4 (5 c.c.), when a blue to blue–green colour will be formed. Another sample is treated with boiling 32% aq. NaOH (4 c.c.), which liberates ammonia from aminoplasts. For identification of the aminoplast, the sample (1 g.) is immersed in boiling N/20 HCl (20 c.c.) for 15 min. The liquor is evaporated to 4 c.c. and 30% formaldehyde (1 c.c.) is added. Drops of the fluid are placed on clean filter-paper and heated 5 min. at 110°C. to reform the resin. Different spots are dyed with each of the following: 1. Crystal Ponceau 6R extra (0·2 g./l., pH 2·0, 5 min. at 20°C.); 2. Neocarmin W (Fesago Chem. Fabrik Dr. Gossler GmbH., Heidelberg; diluted with 3 parts water, 5 min. at 20°C.); 3. A mixture of Crystal Ponceau 6R extra (0·2 g./l.), Crystal Violet (0·2 g./l.), and Celliton Fast Yellow RR (5 g./l.; pH 2·0, 5 min. at 80°C.). The dyed spots were washed in cold water for 30 min. before comparing the colours. A range of 8 commercial resins were examined both on the fibre and as precondensates. The colours obtained are tabulated and illustrated by a colour-photograph. H. E. N.

PATENT

Colour Comparator for Use with Liquids. R. Adler. *BP* 699,181

An opaque casing has an opening in its front wall for horizontal observation and in the rear wall a light transmitting aperture aligned with the viewing opening. The top of the casing has at least one opening through which the lower portion of a tube may be placed between the opening and the aperture. A rotatable disc is mounted in the casing in such position that the peripheral portion of the disc overlaps only part of the two openings. The disc carries a series of colour standards which meet in overlapping contact the tube inserted into the casing. The colour standards are mounted so that the light transmitted through the casing passes through them. C. O. C.

β-Naphthol—Solubility and Diffusivity in Water (IV p. 28).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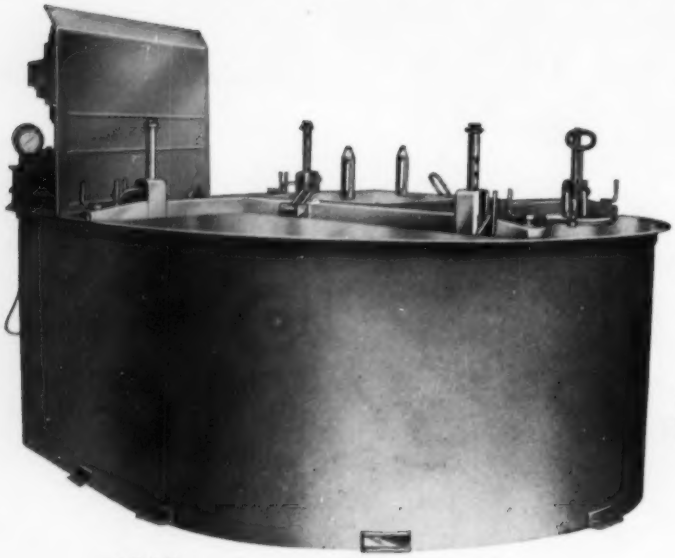

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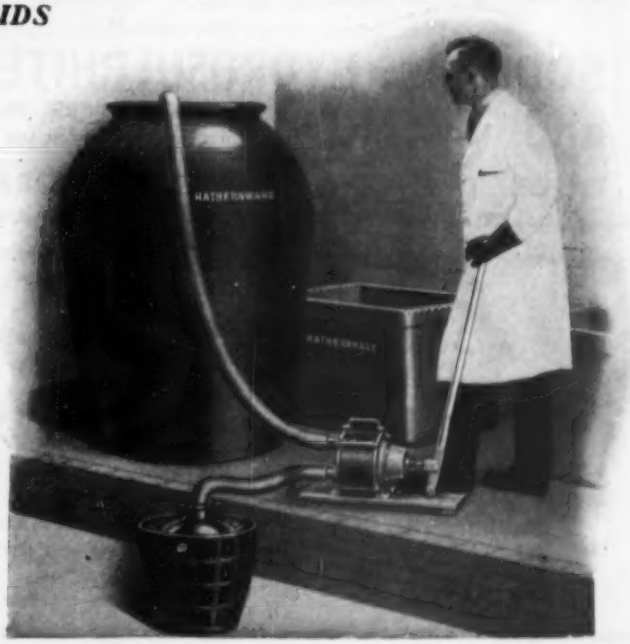

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